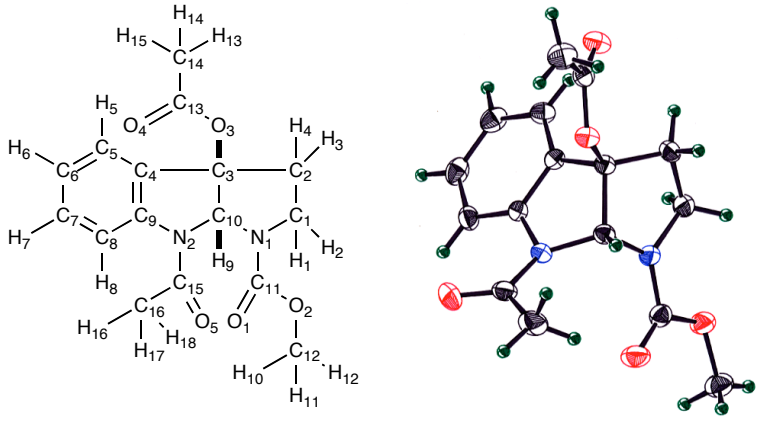
X-Ray single crystal analysis of 40

Figure 2



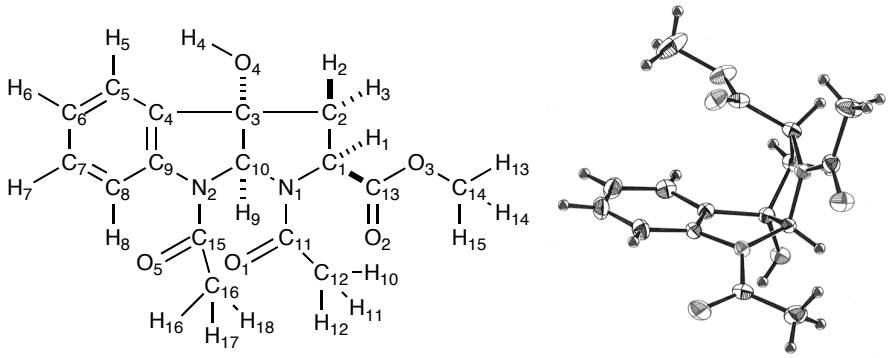
A single crystal (0.30x0.30x0.30 mm) of **40** was obtained by recrystallization from $CHCl_3$-hexane. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Cu-$K\alpha$ radiation (λ=1.54178 Å). Crystal data: $C_{16}H_{18}N_2O_5$, M=318.33, orthorhombic, space group $P2_12_12_1$ (#19), a=12.0955 (9)Å, b=15.231 (1)Å, c=8.4361 (5)Å, V=1554.1 (2)Å^3, Z=4, D_{calc}=1.360 g/cm^3, F(000)=672, and μ(Cu$K\alpha$)=8.12 cm^{-1}. The structure was solved by direct methods using MITHRIL.[18] The non-hydrogen atoms were refined anisotropically. The final cycle of full-matrix least-squares refinement was based on 1265 observed reflections (I>3.00σ (I), 2θ <120.1°) and 280 variable parameters. The final refinement converged with R=0.033 and Rw=0.041.

Table 8. Positional Parameters and *B* (eq) for 40

atom	x	y	z	B (eq)	atom	x	y	z	B (eq)
O (1)	0.7124 (2)	–0.0009 (2)	0.6232 (3)	4.8 (1)	C (15)	0.9343 (3)	0.1224 (2)	0.6971 (4)	3.8 (1)
O (2)	0.5444 (2)	0.0440 (1)	0.5384 (3)	4.1 (1)	C (16)	0.9710 (4)	0.0351 (3)	0.6366 (6)	4.8 (2)
O (3)	0.9129 (2)	0.2341 (1)	0.2879 (2)	3.15 (8)	H (1)	0.572 (3)	0.141 (2)	0.307 (4)	5.17 (3)
O (4)	0.8690 (2)	0.3614 (1)	0.1690 (3)	4.1 (1)	H (2)	0.581 (3)	0.220 (3)	0.427 (4)	4.65 (2)
O (5)	0.9758 (2)	0.1556 (2)	0.8137 (3)	5.8 (1)	H (3)	0.731 (3)	0.180 (2)	0.163 (4)	4.36 (2)
N (1)	0.6920 (2)	0.1186 (1)	0.4650 (3)	3.1 (1)	H (4)	0.683 (3)	0.277 (2)	0.211 (4)	3.94 (2)
N (2)	0.8537 (2)	0.1658 (1)	0.6144 (3)	3.0 (1)	H (5)	0.737 (3)	0.404 (2)	0.401 (4)	4.32 (2)
C (1)	0.6229 (3)	0.1752 (2)	0.3640 (4)	3.6 (1)	H (6)	0.726 (4)	0.487 (3)	0.635 (5)	6.54 (4)
C (2)	0.7073 (3)	0.2196 (2)	0.2571 (4)	3.3 (1)	H (7)	0.789 (3)	0.421 (2)	0.875 (5)	5.24 (2)
C (3)	0.8058 (2)	0.2287 (2)	0.3661 (3)	2.7 (1)	H (8)	0.843 (3)	0.278 (2)	0.878 (5)	4.24 (2)
C (4)	0.7956 (2)	0.2945 (2)	0.4986 (3)	2.7 (1)	H (9)	0.855 (3)	0.095 (2)	0.408 (4)	3.47 (2)
C (5)	0.7594 (3)	0.3808 (2)	0.4963 (4)	3.3 (1)	H (10)	0.430 (4)	–0.032 (3)	0.599 (5)	5.34 (3)
C (6)	0.7560 (3)	0.4268 (2)	0.6367 (5)	4.1 (1)	H (11)	0.495 (7)	–0.015 (3)	0.738 (7)	11.39 (8)
C (7)	0.7879 (3)	0.3878 (2)	0.7768 (4)	4.1 (2)	H (12)	0.540 (3)	–0.081 (3)	0.609 (5)	5.53 (3)
C (8)	0.8233 (3)	0.3014 (2)	0.7817 (4)	3.5 (1)	H (13)	1.091 (3)	0.273 (3)	0.193 (5)	4.95 (2)
C (9)	0.8255 (2)	0.2560 (2)	0.6410 (3)	2.8 (1)	H (14)	1.044 (4)	0.252 (3)	0.058 (5)	6.87 (4)
C (10)	0.8091 (2)	0.1416 (2)	0.4597 (3)	2.8 (1)	H (15)	1.070 (4)	0.356 (3)	0.085 (5)	7.39 (4)
C (11)	0.6552 (3)	0.0488 (2)	0.5482 (4)	3.4 (1)	H (16)	0.911 (3)	–0.001 (3)	0.598 (5)	5.76 (3)
C (12)	0.4946 (4)	–0.0286 (3)	0.6214 (6)	4.7 (2)	H (17)	1.018 (4)	0.011 (3)	0.714 (4)	6.39 (4)
C (13)	0.9351 (3)	0.3044 (2)	0.1957 (3)	3.3 (1)	H (18)	1.011 (4)	0.040 (3)	0.530 (5)	6.32 (3)
C (14)	1.0493 (4)	0.3007 (3)	0.1319 (6)	4.8 (2)					

X-Ray single crystal analysis of 51

Figure 3



A single crystal (0.50x0.30x0.10 mm) of **51** was obtained by recrystallization from MeOH. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Mo-$K\alpha$ radiation (λ=0.71069 Å). Crystal data: $C_{16}H_{18}N_2O_5$, M=318.33, monoclinic, space group $P2_1/a$ (#14), a=8.230 (5)Å, b=20.75 (1)Å, c=9.607 (6)Å, β=112.86 (5)°, V=1512 (2)Å^3, Z=4, D_{calc}=1.398 g/cm^3, F(000)=672, and μ(Mo$K\alpha$)=0.98 cm^{-1}. The structure was solved by direct methods using MITHRIL.[18] The non-hydrogen atoms were refined anisotropically. The final cycle of full-matrix least-squares refinement was based on 1830 observed reflections (I>3.00σ (I), 2θ <55.0°) and 280 variable parameters. The final refinement converged with R=0.045 and Rw=0.050.

Table 9. Positional Parameters and *B* (eq) for 51

atom	x	y	z	B (eq)	atom	x	y	z	B (eq)
O (1)	0.7200 (3)	0.3296 (1)	0.6974 (2)	4.0 (1)	C (15)	0.9996 (4)	0.4208 (1)	0.5978 (3)	2.8 (1)
O (2)	0.5902 (3)	0.2704 (1)	0.2381 (2)	4.0 (1)	C (16)	0.9947 (6)	0.4357 (2)	0.7470 (4)	4.1 (2)
O (3)	0.3547 (3)	0.3165 (1)	0.0607 (2)	4.4 (1)	H (1)	0.322 (4)	0.329 (1)	0.320 (3)	3.2 (6)
O (4)	0.5443 (3)	0.5186 (1)	0.3237 (2)	2.90 (8)	H (2)	0.313 (4)	0.425 (1)	0.147 (3)	3.4 (7)
O (5)	1.1385 (3)	0.4189 (1)	0.5788 (2)	4.4 (1)	H (3)	0.311 (4)	0.442 (1)	0.309 (3)	3.0 (6)
N (1)	0.5701 (3)	0.3564 (1)	0.4557 (2)	2.35 (9)	H (4)	0.636 (5)	0.536 (2)	0.339 (5)	7 (1)
N (2)	0.8447 (3)	0.4095 (1)	0.4767 (2)	2.17 (8)	H (5)	0.518 (4)	0.445 (1)	0.013 (3)	2.7 (6)
C (1)	0.4226 (4)	0.3501 (1)	0.3092 (3)	2.7 (1)	H (6)	0.726 (4)	0.407 (2)	–0.084 (4)	4.1 (8)
C (2)	0.3816 (4)	0.4200 (1)	0.2592 (4)	2.8 (1)	H (7)	0.993 (4)	0.366 (1)	0.076 (3)	3.8 (7)
C (3)	0.5624 (3)	0.4511 (1)	0.3164 (3)	2.3 (1)	H (8)	1.064 (4)	0.366 (1)	0.339 (3)	2.9 (6)
C (4)	0.6719 (3)	0.4306 (1)	0.2281 (3)	2.2 (1)	H (9)	0.663 (3)	0.441 (1)	0.556 (3)	1.2 (5)
C (5)	0.6286 (4)	0.4306 (1)	0.0741 (3)	3.1 (1)	H (10)	0.458 (5)	0.236 (2)	0.464 (5)	7 (1)
C (6)	0.7498 (5)	0.4080 (2)	0.0188 (4)	3.7 (1)	H (11)	0.375 (7)	0.273 (3)	0.555 (6)	11 (2)
C (7)	0.9106 (5)	0.3847 (2)	0.1172 (4)	3.8 (1)	H (12)	0.531 (6)	0.233 (2)	0.651 (5)	10 (1)
C (8)	0.9556 (4)	0.3835 (2)	0.2708 (4)	3.1 (1)	H (13)	0.341 (6)	0.303 (2)	–0.142 (6)	9 (2)
C (9)	0.8337 (3)	0.4071 (1)	0.3249 (3)	2.3 (1)	H (14)	0.338 (8)	0.239 (3)	–0.065 (6)	12 (2)
C (10)	0.6637 (3)	0.4178 (1)	0.4703 (3)	2.2 (1)	H (15)	0.53 (1)	0.271 (4)	–0.019 (8)	17 (3)
C (11)	0.6016 (4)	0.3174 (1)	0.5763 (3)	2.8 (1)	H (16)	0.925 (8)	0.469 (3)	0.753 (6)	12 (2)
C (12)	0.4885 (6)	0.2587 (2)	0.5560 (5)	4.9 (2)	H (17)	1.099 (6)	0.452 (2)	0.809 (5)	8 (1)
C (13)	0.4696 (4)	0.3075 (1)	0.2007 (3)	2.9 (1)	H (18)	0.954 (6)	0.402 (2)	0.791 (5)	9 (1)
C (14)	0.387 (1)	0.2812 (3)	–0.0562 (5)	6.7 (3)					