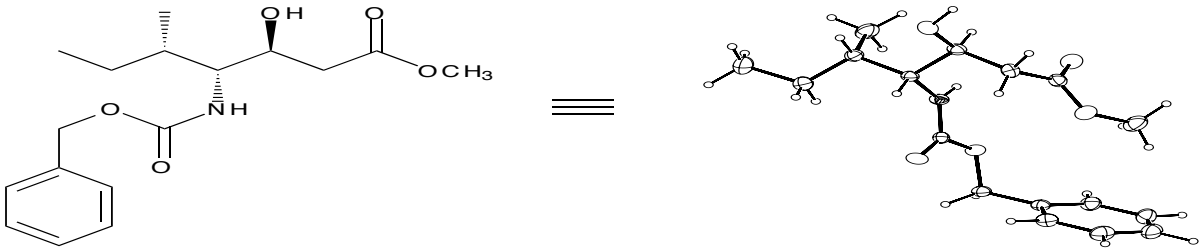
X-ray Structure Determination of Compound 762



Compound 762, $C_{17}H_{25}NO_5$, crystallizes in the orthorhombic space group $P2_12_12_1$ (systematic absences h00: h=odd, 0k0: k=odd, and 00l: l=odd) with a=13.1394(1)Å, b=23.1711(3)Å, c=5.6267(1)Å, V=1713.07(4)Å^3, Z=4 and d_{calc}=1.254 g/cm^3. X-ray intensity data were collected on an Rigaku R-AXIS IIc area detector employing graphite-monochromated Mo-K_α radiation (λ=0.71069 Å) at a temperature of 200°K. Indexing was performed from a series of 1° oscillation images with exposures of 300 seconds per frame. A hemisphere of data was collected using 6° oscillation angles with exposures of 500 seconds per frame and a crystal-to-detector distance of 82 mm. Oscillation images were processed using bioteX[1], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the teXsan[2] program package for further processing and structure solution on a Silicon Graphics Indigo R4000 computer. A total of 11348 reflections were measured over the ranges $3.52 \le 2\theta \le 50.7$ °, $-15 \le h \le 15$, $-27 \le k \le 27$, $-6 \le l \le 6$ yielding 3115 unique reflections (R_{int} = 0.0240). The intensity data were corrected for Lorentz and polarization effects but not for absorption.

The structure was solved by direct methods (SIR92[3]). Refinement was by full-matrix least squares based on F^2 using SHELXL-93[4]. All reflections were used during refinement (F^2's that were experimentally negative were replaced by $F^2 = 0$). The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ 0.0397P^2 + 0.4302P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined isotropically. Refinement converged to R_1=0.0358 and wR_2=0.0814 for 2944 reflections for which $F > 4\sigma(F)$ and R_1=0.0392, wR_2=0.0835 and GOF = 1.044 for all 3115 unique,

non-zero reflections and 309 variables[5]. The maximum Δ/σ in the final cycle of least squares was 0.016 and the two most prominent peaks in the final difference Fourier were +0.173 and -0.176 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Table 2. Anisotropic thermal parameters are in Table 3. Tables 4. and 5. list bond distances and bond angles. Figure 1. is an ORTEP[6] representation of the molecule with 30% probability thermal ellipsoids displayed.

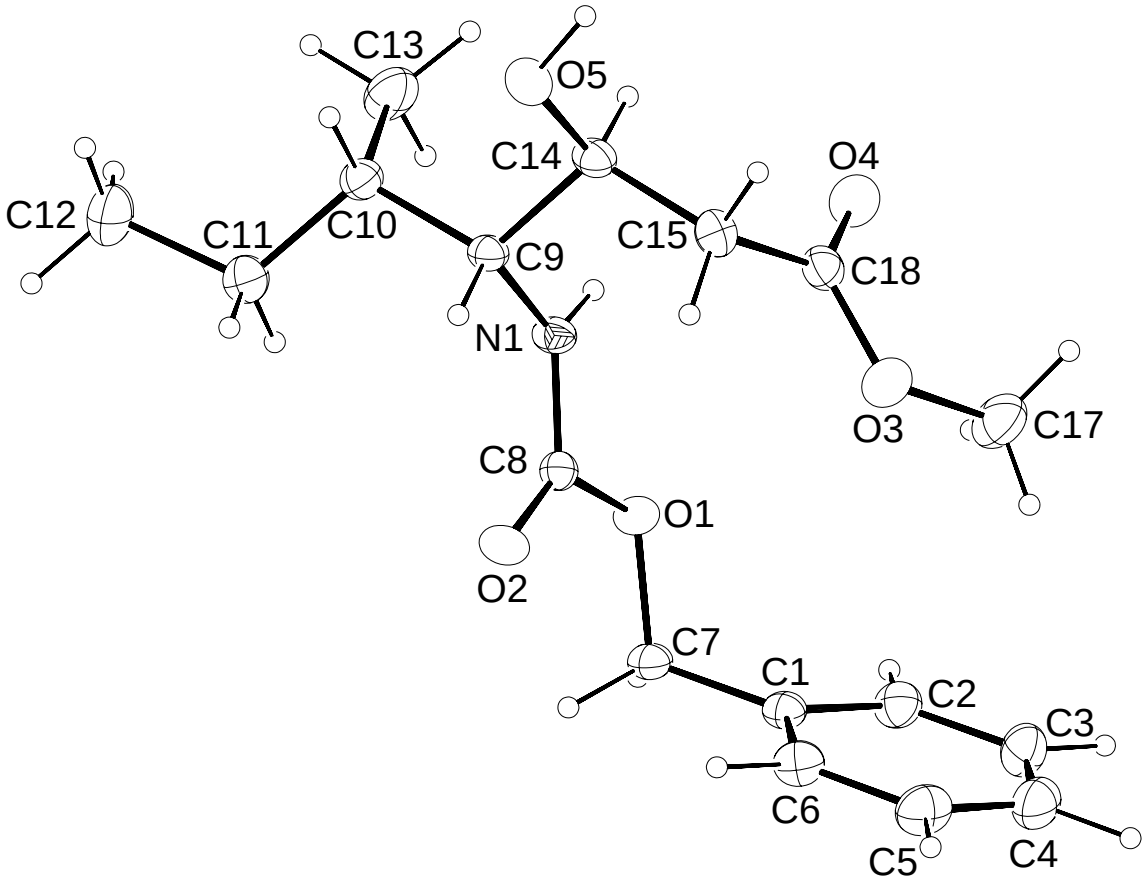


Figure 1. ORTEP drawing of the title compound with 30% probability thermal ellipsoids.

References

[1]. bioteX: A suite of Programs for the Collection, Reduction and Interpretation of Imaging Plate Data, Molecular Structure Corporation (1995).

[2]. teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

[3]. SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidoro, G. (1994). *J. Appl. Cryst.*, **27**, 435.

[4]. SHELXL-93: Program for the Refinement of Crystal Structures, Sheldrick, G.M. (1993), University of Göttingen, Germany.

[5]. $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|$

$wR_2 = \{ \sum w (F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2 \}^{1/2}$

$GOF = \{ \sum w (F_o^2 - F_c^2)^2 / (n - p) \}^{1/2}$

where n = the number of reflections and p = the number of parameters refined.

[6]. "ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.