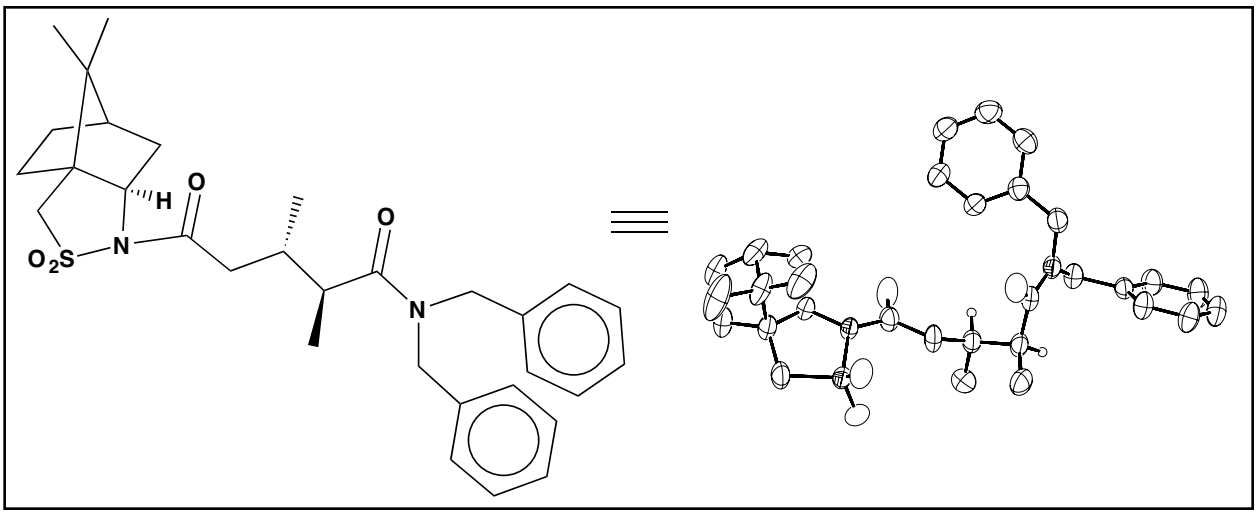
X-ray Structure Determination of Compound 770



Compound 770, $C_{31}H_{40}N_2SO_4$, crystallizes in the triclinic space group P1 with a=9.244(4)Å, b=9.313(8)Å, c=9.120(3)Å, α=93.93(6)°, β=103.63(5)°, γ=103.35(5)°, V=736.1(7)Å^3, Z=1 and d_{calc}=1.211 g/cm^3. The cell constants were determined from a least squares fit of the setting angles for 25 accurately centered reflections. X-ray intensity data were collected on a Rigaku AFC7 diffractometer employing graphite-monochromated Cu-K_α radiation (λ=1.54178Å) at a temperature of 295°K using the ω-2θ scan technique. A total of 2319 reflections were measured over the ranges $9.84 \le 2\theta \le 120.02$ °, $0 \le h \le 10$, $-10 \le k \le 10$, $-10 \le l \le 9$ yielding 2119 unique reflections (R_{int} = 0.7800). The intensity data were corrected for Lorentz and polarization effects but not for absorption.

The structure was solved by direct methods (SIR92[1]). Refinement was by full-matrix least squares based on F^2 using SHELXL-93[2]. All reflections were used during refinement (F^2's that were experimentally negative were replaced by $F^2 = 0$). The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ 0.0650P^2 + 0.1718P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a "riding" model. Refinement converged to R_1=0.0323 and wR_2=0.0853 for 1874 reflections for which $F > 4\sigma(F)$ and R_1=0.0572, wR_2=0.1075 and GOF = 1.033 for all 2119 unique, non-zero reflections and 347 variables[3]. The maximum Δ/σ in the final cycle of least squares was 0.001 and the two most prominent peaks in the final difference Fourier were +0.300 and -0.246 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[4] representation of the molecule with 30% probability thermal ellipsoids displayed.

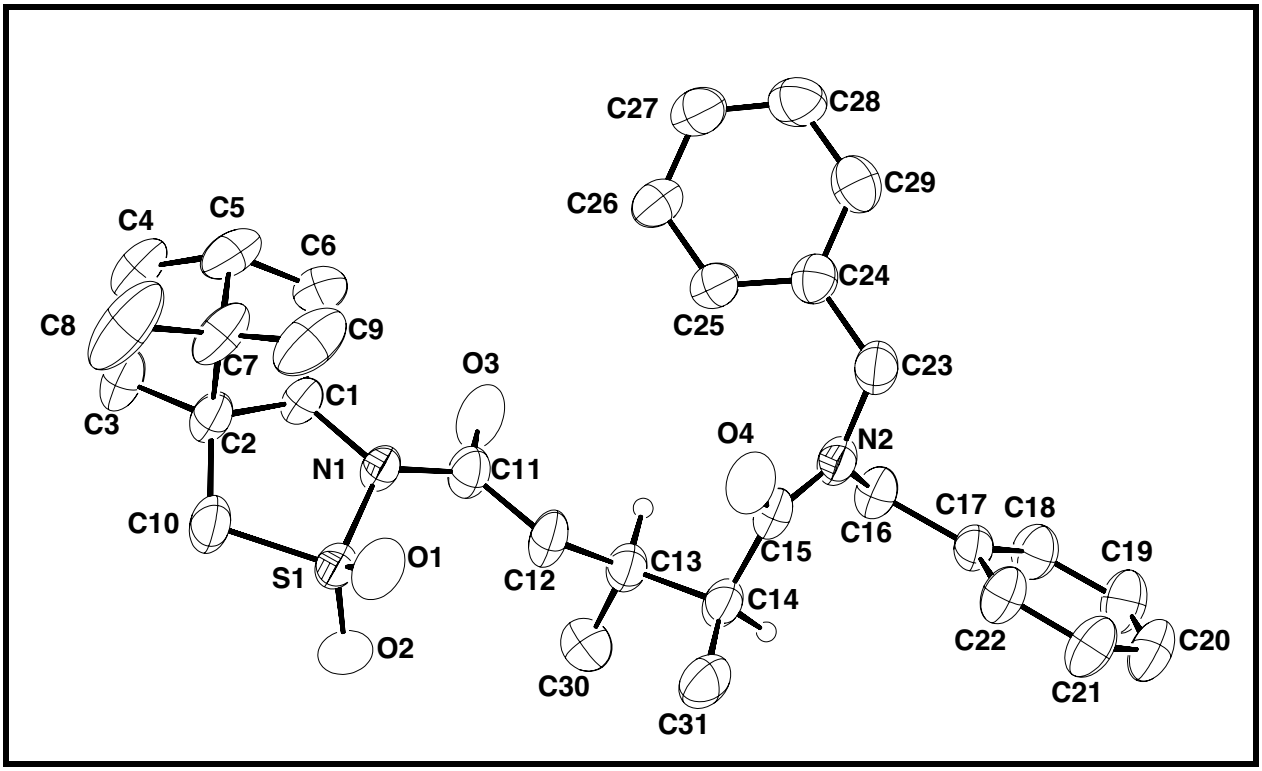


Figure 1. ORTEP drawing of the title compound with 30% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 770

Formula:	**$C_{31}H_{40}N_2SO_4$**
Formula weight:	**536.71**
Crystal class:	**triclinic**
Space group:	**P1 (#1)**
Z	**1**
Cell constants:	
a	**9.244(4)Å**
b	**9.313(8)Å**
c	**9.120(3)Å**

α	93.93(6)°
β	103.63(5)°
γ	103.35(5)°
V	736.1(7)Å^3
μ	12.70 cm^{-1}
crystal size, mm	0.38 x 0.12 x 0.10
D_{calc}	1.211 g/cm^3
F(000)	288
Radiation:	Cu-K_α (λ=1.54178Å)
2θ range	9.84 – 120.02 °
hkl collected:	0≤ h ≤10; -10≤k ≤10; -10≤l ≤9
No. reflections measured:	2319
No. unique reflections:	2119 (R_{int}=0.7800)
No. observed reflections	1874 (F>4σ)
No. reflections used in refinement	2119
No. parameters	347
R indices (F>4σ)	R_1=0.0323
	wR_2=0.0853
R indices (all data)	R_1=0.0572
	wR_2=0.1075
GOF:	1.033
Final Difference Peaks, e/Å^3	+0.300, -0.246

Table 2. Refined Positional Parameters for Compound 770

Atom	x	y	z	U_{eq}, Å^2
S1	0.19825(13)	0.39023(11)	0.75770(12)	0.0744(4)
O1	0.3126(5)	0.3354(4)	0.8550(4)	0.0988(12)
O2	0.0870(4)	0.2864(4)	0.6402(5)	0.1073(13)
O3	0.3392(6)	0.6163(4)	0.4697(4)	0.1054(13)
O4	0.7196(5)	0.3482(4)	0.5362(4)	0.0921(10)
N1	0.2823(4)	0.5354(3)	0.6800(4)	0.0612(8)
N2	0.7228(4)	0.3991(4)	0.3007(4)	0.0691(9)
C1	0.2346(5)	0.6716(4)	0.7141(5)	0.0627(10)
H1	0.1504(5)	0.6798(4)	0.6294(5)	0.083
C2	0.1771(6)	0.6572(5)	0.8588(5)	0.0730(12)
C3	0.0724(8)	0.7640(6)	0.8541(7)	0.097(2)
H3a	-0.0020(8)	0.7486(6)	0.7560(7)	0.129
H3b	0.0183(8)	0.7517(6)	0.9333(7)	0.129
C4	0.1862(8)	0.9176(6)	0.8813(6)	0.100(2)
H4a	0.1671(8)	0.9703(6)	0.7935(6)	0.133
H4b	0.1800(8)	0.9775(6)	0.9698(6)	0.133
C5	0.3416(8)	0.8809(6)	0.9071(7)	0.101(2)

H5	0.4286(8)	0.9630(6)	0.9630(7)	0.135
C6	0.3582(7)	0.8196(5)	0.7547(7)	0.093(2)
H6a	0.4601(7)	0.8040(5)	0.7640(7)	0.123
H6b	0.3391(7)	0.8859(5)	0.6790(7)	0.123
C7	0.3199(8)	0.7425(6)	0.9876(6)	0.101(2)
C8	0.278(2)	0.7650(11)	1.1410(7)	0.180(5)
H8a	0.367(3)	0.822(10)	1.217(3)	0.270
H8b	0.241(11)	0.6698(11)	1.172(6)	0.270
H8c	0.199(9)	0.818(10)	1.129(3)	0.270
C9	0.4589(9)	0.6728(8)	1.0176(8)	0.137(3)
H9a	0.433(3)	0.583(4)	1.062(7)	0.205
H9b	0.546(2)	0.742(3)	1.086(6)	0.205
H9c	0.483(5)	0.651(7)	0.9234(14)	0.205
C10	0.1042(8)	0.4950(6)	0.8593(8)	0.104(2)
H10a	-0.0051(8)	0.4714(6)	0.8100(8)	0.139
H10b	0.1174(8)	0.4716(6)	0.9629(8)	0.139
C11	0.3315(6)	0.5168(5)	0.5492(5)	0.0738(12)
C12	0.3850(7)	0.3797(6)	0.5217(5)	0.0808(13)
H12a	0.3202(7)	0.2969(6)	0.5540(5)	0.108
H12b	0.4893(7)	0.3946(6)	0.5842(5)	0.108
C13	0.3823(5)	0.3385(5)	0.3554(5)	0.0701(12)
H13	0.4233(5)	0.4305(5)	0.3164(5)	0.093
C14	0.4900(6)	0.2342(5)	0.3451(5)	0.0720(12)
H14	0.4720(6)	0.1964(5)	0.2377(5)	0.096
C15	0.6531(6)	0.3271(5)	0.4001(5)	0.0707(12)
C16	0.6628(6)	0.3753(5)	0.1352(5)	0.0720(12)
H16a	0.6918(6)	0.4689(5)	0.0959(5)	0.096
H16b	0.5513(6)	0.3445(5)	0.1104(5)	0.096
C17	0.7204(5)	0.2594(5)	0.0567(5)	0.0624(10)
C18	0.7186(6)	0.2615(6)	-0.0950(5)	0.0793(13)
H18	0.6826(6)	0.3339(6)	-0.1472(5)	0.105

C19	0.7696(8)	0.1575(7)	-0.1698(6)	0.098(2)
H19	0.7708(8)	0.1619(7)	-0.2711(6)	0.130
C20	0.8185(7)	0.0478(7)	-0.0959(6)	0.097(2)
H20	0.8498(7)	-0.0244(7)	-0.1473(6)	0.129
C21	0.8209(7)	0.0450(6)	0.0543(6)	0.093(2)
H21	0.8547(7)	-0.0292(6)	0.1052(6)	0.124
C22	0.7739(6)	0.1506(5)	0.1311(5)	0.0795(13)
H22	0.7782(6)	0.1485(5)	0.2339(5)	0.106
C23	0.8745(6)	0.5027(6)	0.3591(6)	0.0834(14)
H23a	0.9330(6)	0.5001(6)	0.2840(6)	0.111
H23b	0.9289(6)	0.4703(6)	0.4502(6)	0.111
C24	0.8668(6)	0.6614(5)	0.3965(5)	0.0716(12)
C25	0.7452(6)	0.6940(5)	0.4413(6)	0.0854(14)
H25	0.6612(6)	0.6166(5)	0.4417(6)	0.114
C26	0.7435(7)	0.8387(5)	0.4859(6)	0.088(2)
H26	0.6589(7)	0.8568(5)	0.5151(6)	0.117
C27	0.8644(7)	0.9543(6)	0.4872(6)	0.091(2)
H27	0.8635(7)	1.0513(6)	0.5182(6)	0.121
C28	0.9867(8)	0.9265(6)	0.4427(6)	0.097(2)
H28	1.0698(8)	1.0052(6)	0.4433(6)	0.129
C29	0.9892(7)	0.7821(7)	0.3963(6)	0.089(2)
H29	1.0732(7)	0.7654(7)	0.3647(6)	0.119
C30	0.2175(6)	0.2709(6)	0.2576(7)	0.093(2)
H30a	0.156(2)	0.341(2)	0.264(4)	0.139
H30b	0.174(2)	0.181(3)	0.294(3)	0.139
H30c	0.2191(8)	0.249(5)	0.1537(11)	0.139

C31	0.4623(8)	0.1012(6)	0.4322(8)	0.103(2)
H31a	0.3553(14)	0.049(3)	0.401(4)	0.155
H31b	0.491(6)	0.1351(8)	0.5394(8)	0.155
H31c	0.523(5)	0.036(3)	0.411(5)	0.155

$$U_{eq}=1/3[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$$

Table 3. Refined Thermal Parameters (U's) for Compound 770

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	0.0925(8)	0.0586(6)	0.0849(7)	0.0067(5)	0.0459(7)	0.0218(6)
O1	0.143(3)	0.089(2)	0.092(2)	0.032(2)	0.045(2)	0.064(2)
O2	0.099(3)	0.067(2)	0.147(3)	-0.017(2)	0.040(3)	0.003(2)
O3	0.172(4)	0.096(2)	0.094(2)	0.037(2)	0.079(3)	0.071(3)
O4	0.105(3)	0.116(3)	0.063(2)	0.005(2)	0.020(2)	0.047(2)
N1	0.080(2)	0.057(2)	0.057(2)	0.0075(14)	0.027(2)	0.028(2)
N2	0.073(2)	0.073(2)	0.067(2)	-0.004(2)	0.024(2)	0.027(2)
C1	0.080(3)	0.058(2)	0.058(2)	0.006(2)	0.021(2)	0.030(2)
C2	0.101(3)	0.067(3)	0.066(3)	0.009(2)	0.036(2)	0.036(2)
C3	0.124(5)	0.097(4)	0.097(4)	0.009(3)	0.045(3)	0.064(4)
C4	0.148(6)	0.078(3)	0.085(3)	-0.002(3)	0.024(3)	0.060(4)
C5	0.120(5)	0.071(3)	0.099(4)	-0.020(3)	0.003(3)	0.031(3)
C6	0.101(4)	0.062(3)	0.115(4)	0.006(3)	0.032(3)	0.018(3)
C7	0.145(5)	0.096(4)	0.065(3)	-0.011(3)	0.006(3)	0.066(4)
C8	0.338(14)	0.176(7)	0.059(3)	-0.003(4)	0.041(5)	0.149(9)
C9	0.154(7)	0.126(5)	0.107(5)	-0.029(4)	-0.036(5)	0.073(5)
C10	0.141(5)	0.085(3)	0.125(4)	0.019(3)	0.092(4)	0.045(3)
C11	0.095(3)	0.076(3)	0.067(3)	0.015(2)	0.038(2)	0.033(2)
C12	0.106(4)	0.088(3)	0.073(3)	0.011(2)	0.046(3)	0.049(3)
C13	0.083(3)	0.067(3)	0.071(3)	0.008(2)	0.035(2)	0.027(2)
C14	0.089(3)	0.069(3)	0.068(2)	-0.001(2)	0.034(2)	0.028(2)
C15	0.085(3)	0.074(3)	0.066(3)	0.001(2)	0.030(3)	0.036(2)
C16	0.086(3)	0.075(3)	0.068(2)	0.011(2)	0.026(2)	0.038(2)
C17	0.063(2)	0.067(2)	0.061(2)	0.003(2)	0.023(2)	0.020(2)
C18	0.096(3)	0.091(3)	0.059(3)	0.012(2)	0.027(2)	0.034(3)
C19	0.120(5)	0.124(4)	0.064(3)	0.000(3)	0.039(3)	0.045(4)
C20	0.108(4)	0.113(4)	0.076(3)	-0.015(3)	0.020(3)	0.054(4)
C21	0.121(4)	0.095(3)	0.077(3)	0.009(3)	0.018(3)	0.063(3)
C22	0.104(4)	0.088(3)	0.061(2)	0.011(2)	0.025(2)	0.049(3)
C23	0.074(3)	0.093(3)	0.087(3)	-0.003(3)	0.028(3)	0.027(3)
C24	0.074(3)	0.080(3)	0.060(2)	0.005(2)	0.015(2)	0.022(2)
C25	0.078(3)	0.069(3)	0.105(4)	-0.007(2)	0.024(3)	0.014(2)
C26	0.102(4)	0.067(3)	0.096(3)	-0.002(2)	0.028(3)	0.025(3)
C27	0.111(4)	0.072(3)	0.086(3)	0.002(2)	0.025(3)	0.018(3)
C28	0.118(5)	0.077(3)	0.094(4)	0.019(3)	0.036(3)	0.010(3)
C29	0.092(4)	0.108(4)	0.075(3)	0.023(3)	0.034(3)	0.025(3)
C30	0.082(3)	0.095(4)	0.103(4)	0.013(3)	0.029(3)	0.019(3)
C31	0.130(5)	0.071(3)	0.129(5)	0.015(3)	0.055(4)	0.043(3)

The form of the anisotropic displacement parameter is:

$$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)].$$

Table 4. Bond Distances in Compound 770, Å

S1-O2	1.422(4)	S1-O1	1.427(4)	S1-N1	1.685(4)
S1-C10	1.792(5)	O3-C11	1.215(6)	O4-C15	1.228(5)
N1-C11	1.387(5)	N1-C1	1.473(5)	N2-C15	1.359(6)
N2-C16	1.461(6)	N2-C23	1.461(6)	C1-C6	1.531(7)
C1-C2	1.538(6)	C2-C10	1.507(8)	C2-C3	1.535(7)
C2-C7	1.552(8)	C3-C4	1.532(9)	C4-C5	1.522(9)
C5-C6	1.521(8)	C5-C7	1.523(9)	C7-C9	1.546(9)
C7-C8	1.552(9)	C11-C12	1.498(7)	C12-C13	1.532(6)
C13-C30	1.534(7)	C13-C14	1.556(6)	C14-C15	1.502(7)
C14-C31	1.522(8)	C16-C17	1.516(6)	C17-C18	1.382(6)
C17-C22	1.378(7)	C18-C19	1.378(7)	C19-C20	1.366(8)
C20-C21	1.367(7)	C21-C22	1.376(7)	C23-C24	1.516(7)
C24-C25	1.371(7)	C24-C29	1.399(7)	C25-C26	1.385(7)
C26-C27	1.358(8)	C27-C28	1.359(9)	C28-C29	1.389(8)

Table 5. Bond Angles in Compound 770, °

O2-S1-O1	117.2(2)	O2-S1-N1	108.9(2)	O1-S1-N1	110.2(2)
O2-S1-C10	109.9(3)	O1-S1-C10	113.3(3)	N1-S1-C10	95.1(2)
C11-N1-C1	119.0(3)	C11-N1-S1	122.3(3)	C1-N1-S1	112.9(3)
C15-N2-C16	125.3(4)	C15-N2-C23	118.9(4)	C16-N2-C23	115.7(4)
N1-C1-C6	118.0(4)	N1-C1-C2	107.5(3)	C6-C1-C2	103.0(4)
C10-C2-C3	116.8(5)	C10-C2-C1	107.8(4)	C3-C2-C1	105.2(4)
C10-C2-C7	120.7(5)	C3-C2-C7	101.1(4)	C1-C2-C7	103.5(4)
C4-C3-C2	103.0(5)	C5-C4-C3	103.1(4)	C6-C5-C4	108.4(5)
C6-C5-C7	102.0(4)	C4-C5-C7	103.4(6)	C5-C6-C1	102.8(5)
C5-C7-C9	114.6(7)	C5-C7-C8	114.4(5)	C9-C7-C8	107.4(6)
C5-C7-C2	92.6(4)	C9-C7-C2	116.2(4)	C8-C7-C2	111.5(7)
C2-C10-S1	107.1(4)	O3-C11-N1	118.7(4)	O3-C11-C12	123.7(4)
N1-C11-C12	117.4(4)	C11-C12-C13	113.9(4)	C12-C13-C30	111.4(4)
C12-C13-C14	110.2(4)	C30-C13-C14	112.3(4)	C15-C14-C31	111.4(5)
C15-C14-C13	107.6(3)	C31-C14-C13	113.8(4)	O4-C15-N2	119.6(5)
O4-C15-C14	120.4(5)	N2-C15-C14	119.7(4)	N2-C16-C17	113.6(3)
C18-C17-C22	118.4(4)	C18-C17-C16	119.2(4)	C22-C17-C16	122.3(4)
C19-C18-C17	120.7(5)	C20-C19-C18	120.3(5)	C21-C20-C19	119.4(5)
C20-C21-C22	120.8(5)	C21-C22-C17	120.4(4)	N2-C23-C24	113.0(4)
C25-C24-C29	116.7(5)	C25-C24-C23	122.1(5)	C29-C24-C23	121.1(5)
C24-C25-C26	122.0(5)	C27-C26-C25	120.6(5)	C28-C27-C26	119.1(5)
C27-C28-C29	120.9(6)	C28-C29-C24	120.8(6)		

References

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

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

3. $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|$

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

   $GOF = \{ \sum w (F_o^2 - F_c^2)^2 / (n - p) \}^{1/2}$
   where n = the number of reflections and p = the number of parameters refined.

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