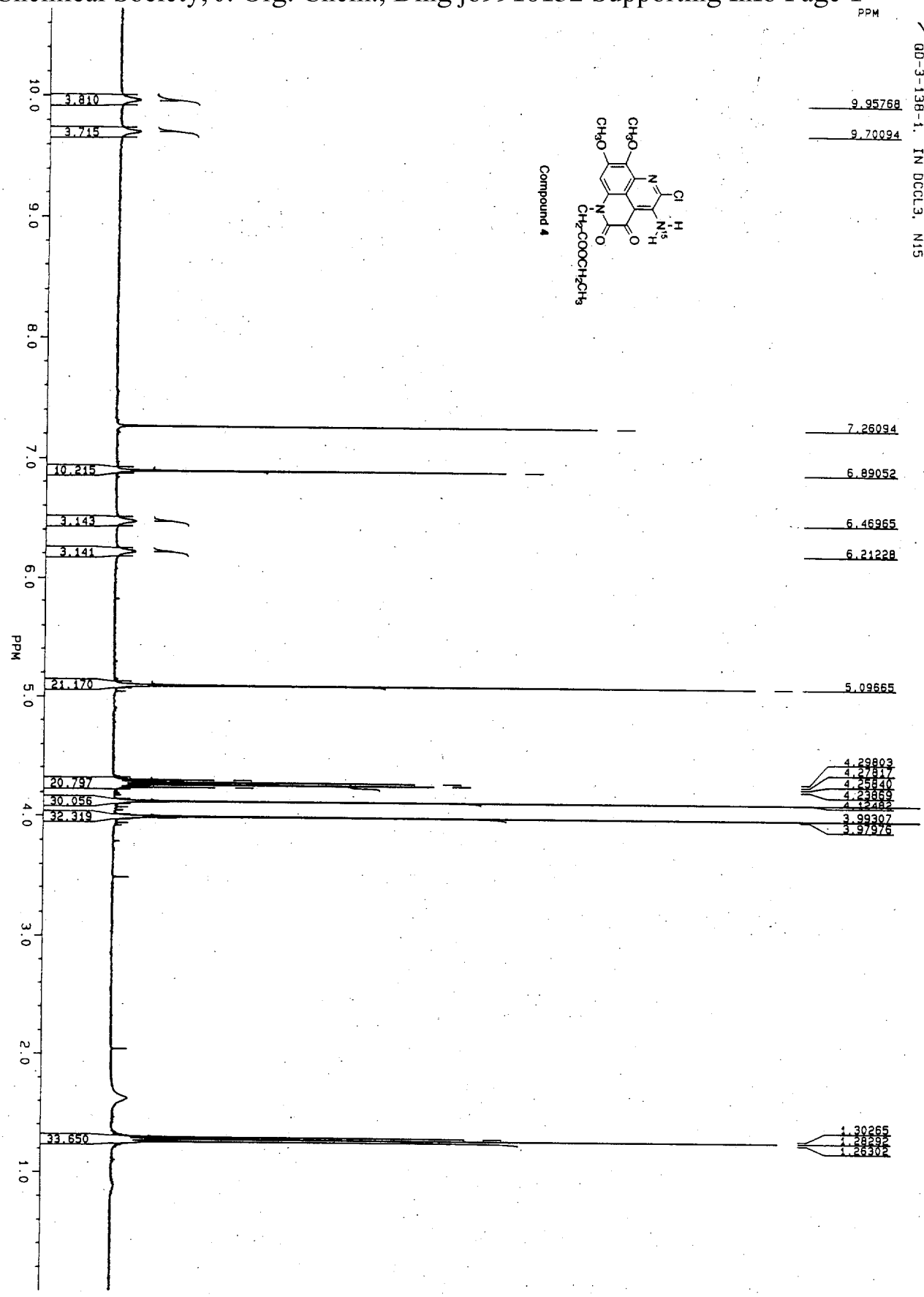

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TD 32768
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AQ 2.720
RG 100
NS 16
TE 297
FW 7600
O2 0.0
DP 40L PO
LB 0.0
GB 0.0
CX 36.00
CY 0.0
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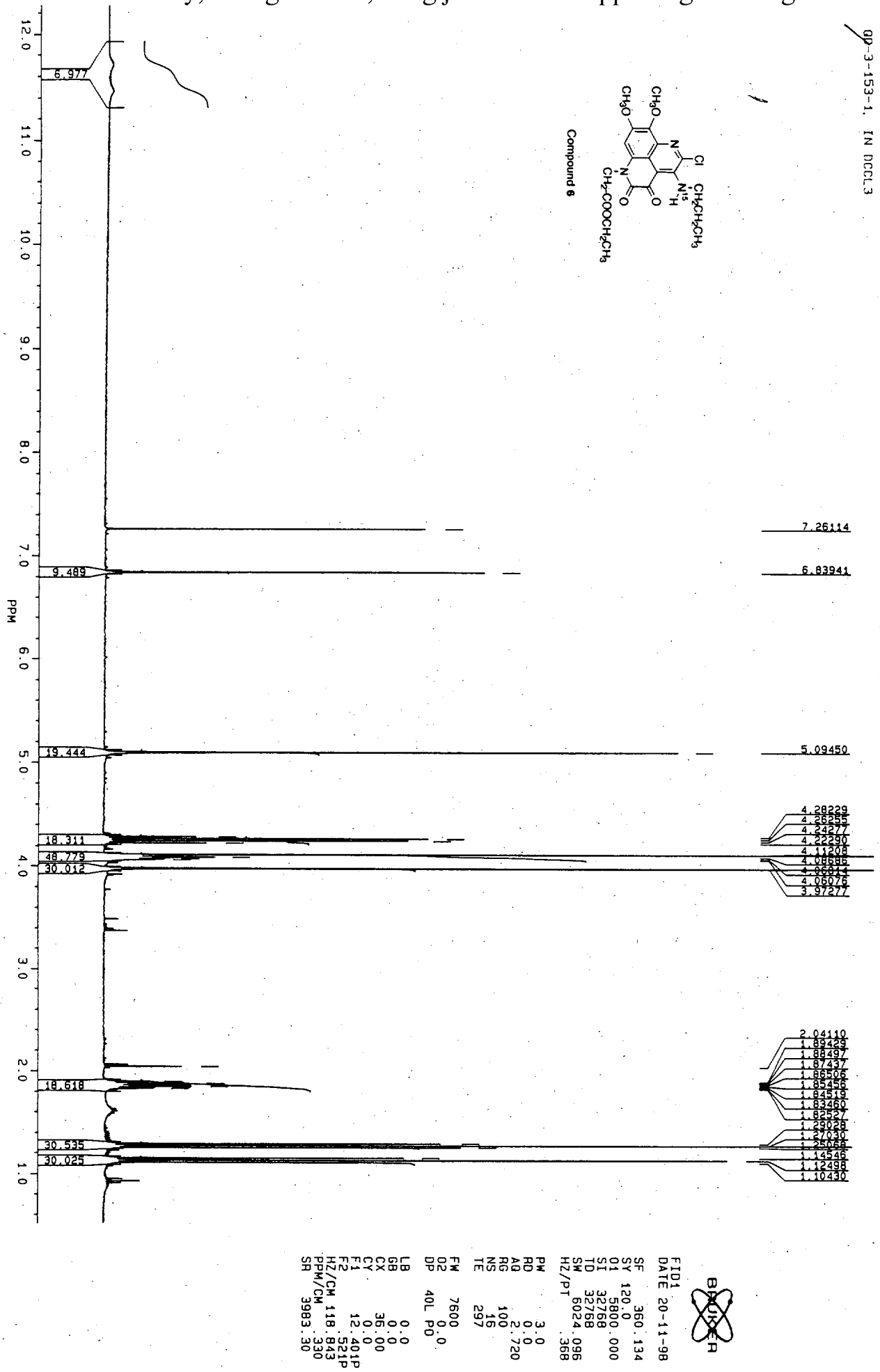
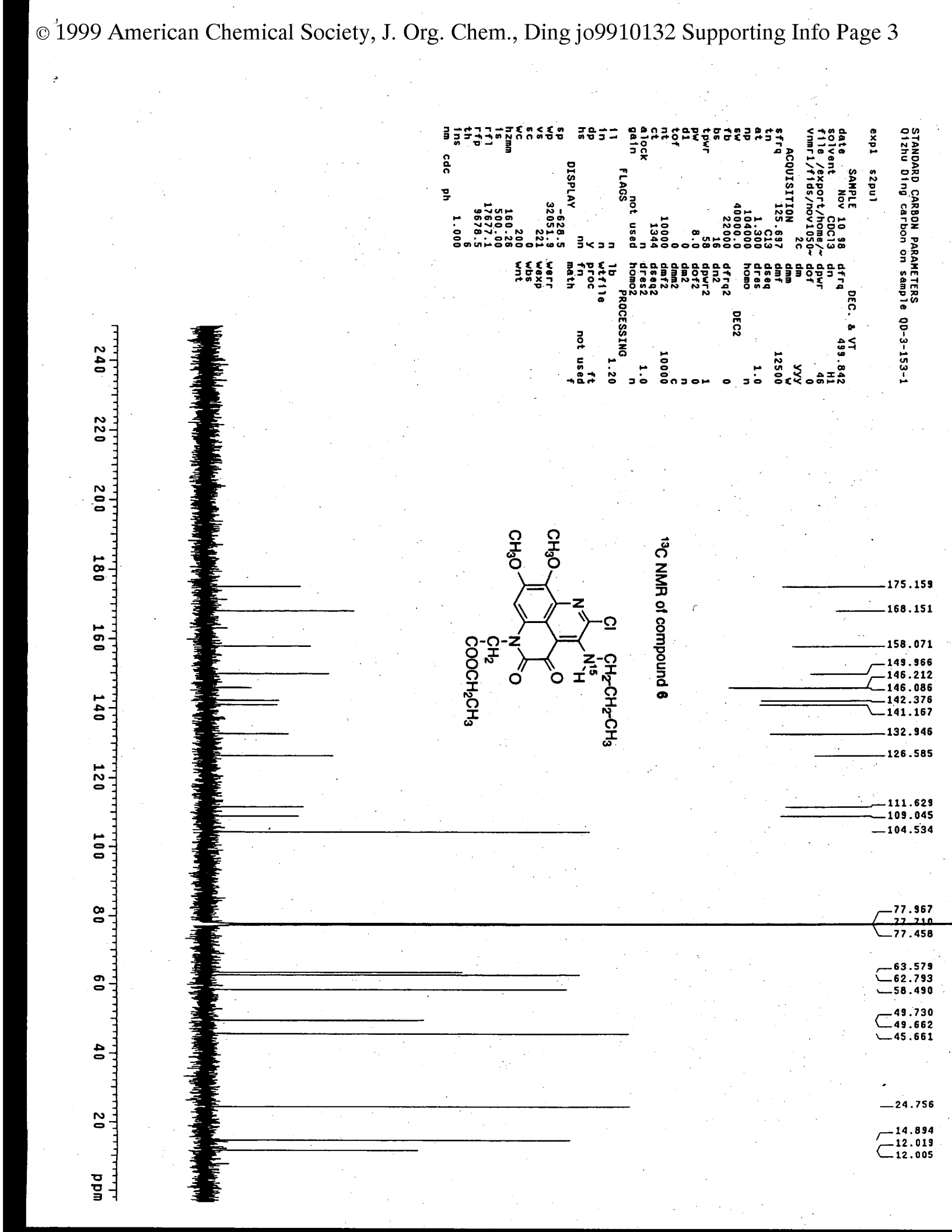
F2 .001P
HZ/CM 108.037
PPM/CM .300
SR 3983.30





University of Alberta Department of Chemistry
Structure Determination Laboratory

STRUCTURE REPORT

SDL Code: JWL9906
Date: 7 April 1999
Compound: 2-Chloro-8,9-dimethoxy-3-(3,4-dimethoxyphenyl)amino-6-ethoxycarbonylmethyl-benzo[*ij*][2,7]naphthyridin-4,5-dione
Formula: $C_{25}H_{24}ClN_3O_8$
Supervisor: J. W. Lown

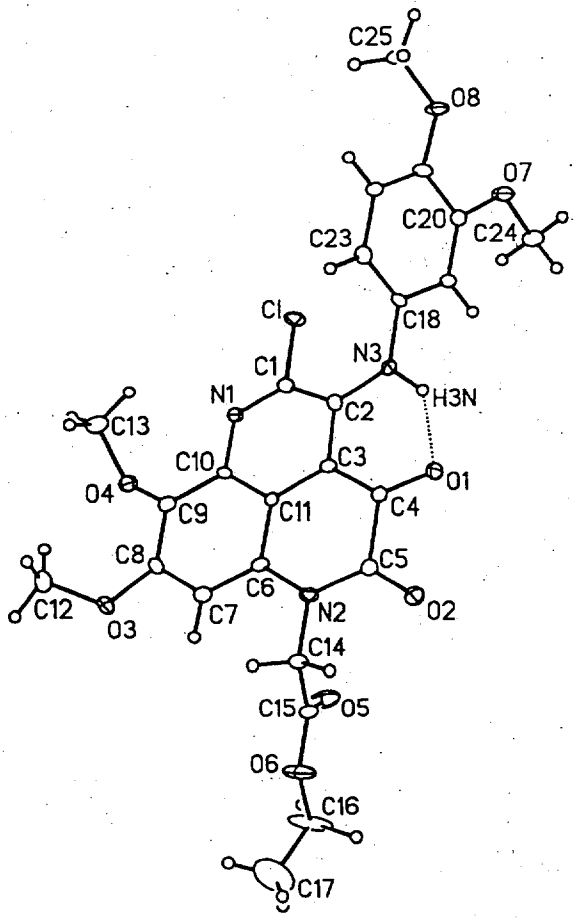
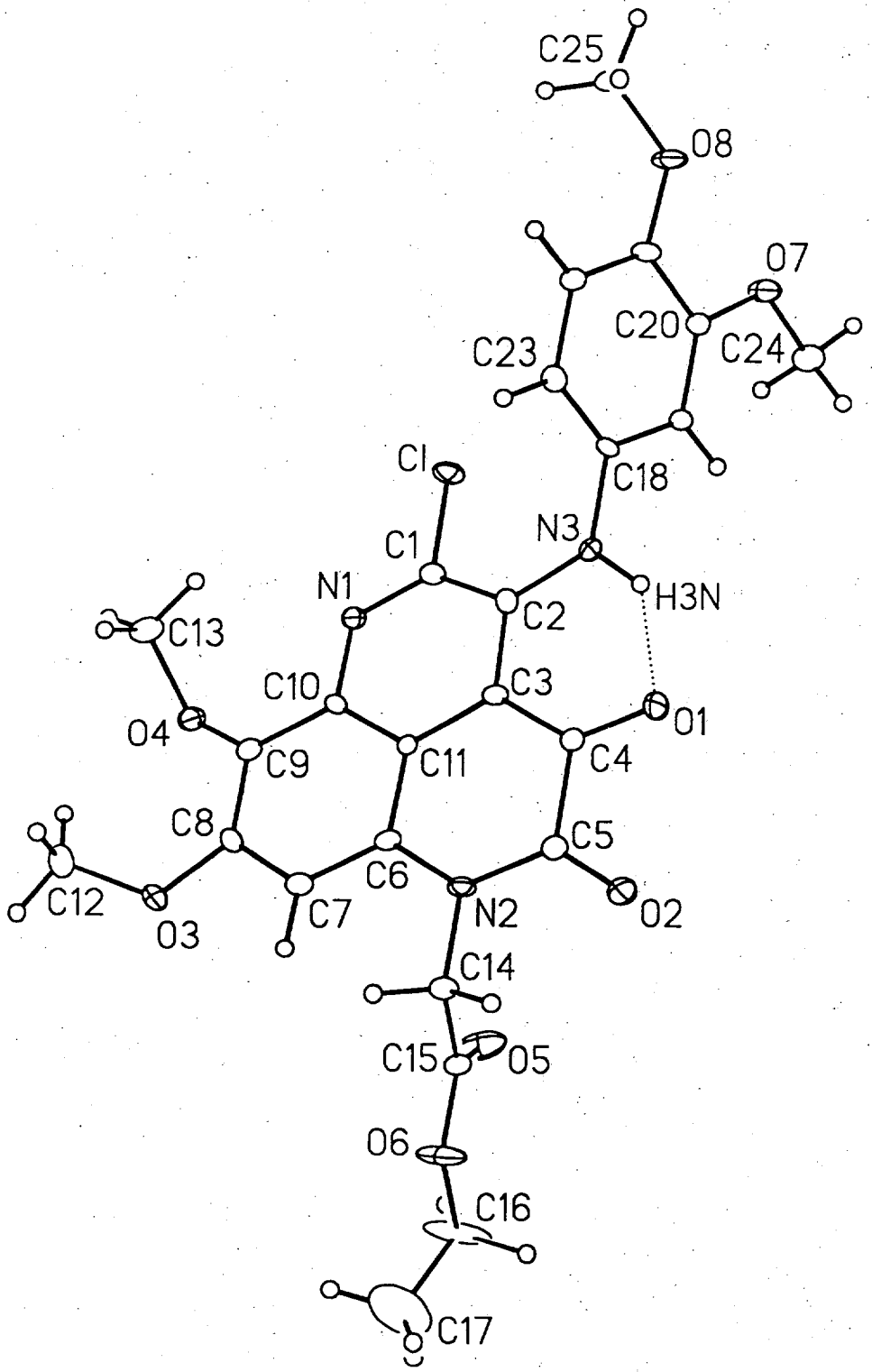
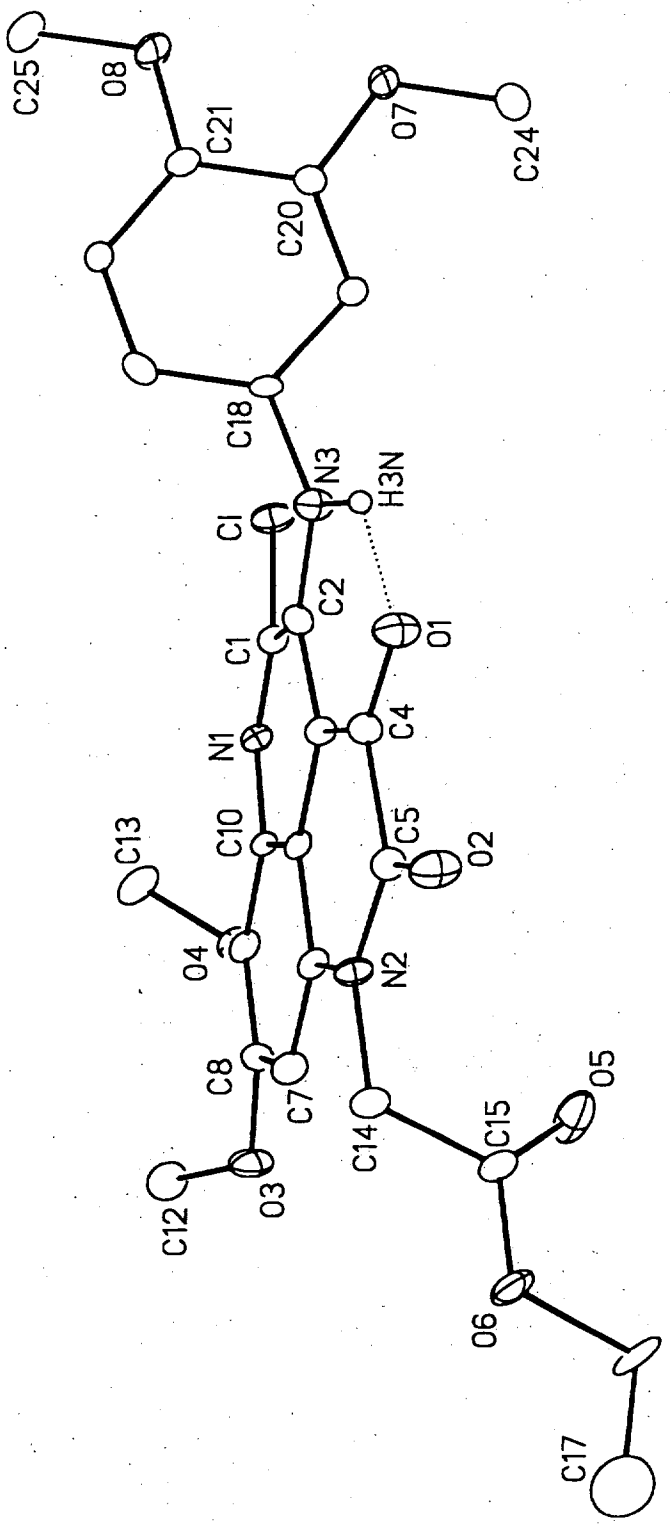


Figure Legends

Figure 1. Perspective view of the 2-chloro-8,9-dimethoxy-3-(3,4-dimethoxyphenyl)amino-6-ethoxycarbonylmethyl-benzo[*ij*][2,7]naphthyridin-4,5-dione molecule showing the atom labelling scheme. Non-hydrogen atoms are represented by Gaussian ellipsoids at the 20% probability level. Hydrogen atoms are shown with arbitrarily small thermal parameters.

Figure 2. Alternate view of the molecule. All hydrogens except for that bound to N3 have been omitted.




C25
O8
C21
O7
C24
C20
C18
N3
H3N
Cl
C2
O1
C1
C4
N1
C10
C5
O2
C13
O4
N2
C8
C7
C14
C15
O5
O3
C12
O6
C17

List of Tables

Table 1. Crystallographic Experimental Details

A. Crystal Data	
formula	$C_{25}H_{24}ClN_3O_8$
formula weight	529.92
crystal dimensions (mm)	0.44 × 0.07 × 0.02
crystal system	orthorhombic
space group	*Pbca* (No. 61)
unit cell parameters[a]	
a (Å)	21.437 (2)
b (Å)	7.5299 (7)
c (Å)	29.734 (3)
V (Å^3)	4799.5 (9)
Z	8
ρ_{calcd} (g cm^{-3})	1.467
μ (mm^{-1})	0.217
B. Data Collection and Refinement Conditions	
diffractometer	Bruker P4/RA/SMART 1000 CCD[b]
radiation (λ [Å])	graphite-monochromated Mo Kα (0.71073)
temperature (°C)	–80
scan type	ϕ rotations (0.3°) / ω scans (0.3°) (30 s exposures)
data collection 2θ limit (deg)	51.70
total data collected	23669 ($-26 \le h \le 26$, $-8 \le k \le 9$, $-36 \le l \le 36$)
independent reflections	4570
number of observations (*NO*)	1511 [$F_o^2 \ge 2\sigma(F_o^2)$]
structure solution method	direct methods/fragment search (*DIRDIF–96*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–93*[d])
absorption correction method	*SADABS*
range of transmission factors	0.9956–0.9829
data/restraints/parameters	4570 [$F_o^2 \ge -3\sigma(F_o^2)$] / 0 / 334
goodness-of-fit (S)[e]	0.777 [$F_o^2 \ge -3\sigma(F_o^2)$]
final R indices[f]	
R_1 [$F_o^2 \ge 2\sigma(F_o^2)$]	0.0570
wR_2 [$F_o^2 \ge -3\sigma(F_o^2)$]	0.1296
largest difference peak and hole	0.233 and –0.397 e Å^{-3}

[a]Obtained from least-squares refinement of 2715 centered reflections.

[b]Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker.

(continued)

Table 1. Crystallographic Experimental Details (continued)

[c]Beurskens, P. T.; Beurskens, G.; Bosman, W. P.; de Gelder, R.; Garcia Granda, S.; Gould, R. O.; Israel, R.; Smits, J. M. M. (1996). The *DIRDIF-96* program system. Crystallography Laboratory, University of Nijmegen, The Netherlands.

[d]Sheldrick, G. M. *SHELXL-93*. Program for crystal structure determination. University of Göttingen, Germany, 1993. Refinement on F_o^2 for all reflections (all of these having $F_o^2 \geq -3\sigma(F_o^2)$). Weighted R-factors wR_2 and all goodnesses of fit S are based on F_o^2; conventional R-factors R_1 are based on F_o, with F_o set to zero for negative F_o^2. The observed criterion of $F_o^2 > 2\sigma(F_o^2)$ is used only for calculating R_1, and is not relevant to the choice of reflections for refinement. R-factors based on F_o^2 are statistically about twice as large as those based on F_o, and R-factors based on ALL data will be even larger.

[e]$S = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.0378P)^2]^{-1}$ where $P = [\mathrm{Max}(F_o^2, 0) + 2F_c^2]/3$).

[f]$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters

Atom	*x*	*y*	*z*	U_{eq}, Å^2
Cl	0.22606(6)	0.23920(16)	0.12929(4)	0.0391(4)*
O1	0.05010(15)	-0.0008(4)	0.24974(11)	0.0403(10)*
O2	0.05738(16)	-0.1083(5)	0.33539(11)	0.0450(10)*
O3	0.38366(17)	-0.0353(4)	0.33833(11)	0.0457(10)*
O4	0.38524(14)	0.1101(4)	0.25005(11)	0.0341(9)*
O5	0.17772(19)	0.1311(4)	0.40487(12)	0.0655(13)*
O6	0.17436(19)	-0.0879(4)	0.45461(12)	0.0644(13)*
O7	0.02278(16)	0.3755(4)	0.04087(10)	0.0452(10)*
O8	0.06407(16)	0.1262(4)	-0.01042(11)	0.0455(10)*
N1	0.27321(17)	0.1195(4)	0.20239(12)	0.0250(10)*
N2	0.16358(19)	-0.1041(5)	0.33367(12)	0.0291(10)*
N3	0.10645(18)	0.0846(5)	0.17410(13)	0.0341(11)*
C1	0.2206(2)	0.1304(6)	0.18166(15)	0.0271(12)*
C2	0.1611(2)	0.0743(6)	0.19745(16)	0.0294(13)*
C3	0.1604(2)	0.0205(5)	0.24303(16)	0.0251(12)*
C4	0.1030(2)	-0.0181(6)	0.26522(16)	0.0295(13)*
C5	0.1055(3)	-0.0802(6)	0.31528(17)	0.0308(13)*
C6	0.2200(2)	-0.0604(5)	0.31169(15)	0.0242(12)*
C7	0.2768(2)	-0.0768(6)	0.33243(16)	0.0300(12)*
C8	0.3323(2)	-0.0271(6)	0.31114(17)	0.0291(13)*
C9	0.3318(2)	0.0366(6)	0.26714(16)	0.0284(13)*
C10	0.2737(2)	0.0486(5)	0.24551(15)	0.0228(11)*
C11	0.2171(2)	0.0013(5)	0.26645(14)	0.0207(11)*
C12	0.4418(2)	-0.0886(7)	0.32186(18)	0.0610(19)*
C13	0.4088(2)	0.0404(7)	0.20813(16)	0.0519(16)*
C14	0.1650(2)	-0.1702(6)	0.37965(14)	0.0302(12)*
C15	0.1737(2)	-0.0218(7)	0.41353(17)	0.0335(13)*
C16	0.1795(4)	0.0443(9)	0.4907(2)	0.129(4)*
C17	0.2091(4)	-0.0174(9)	0.5262(2)	0.134(4)*
C18	0.0971(2)	0.0894(6)	0.12615(16)	0.0257(12)*
C19	0.0636(2)	0.2333(6)	0.10837(15)	0.0287(12)*
C20	0.0540(2)	0.2408(7)	0.06257(16)	0.0308(12)*
C21	0.0760(2)	0.1047(6)	0.03446(16)	0.0327(13)*
C22	0.1084(2)	-0.0363(6)	0.05324(16)	0.0306(13)*
C23	0.1184(2)	-0.0447(6)	0.09913(17)	0.0342(14)*
C24	0.0064(2)	0.5287(6)	0.06708(16)	0.0479(16)*
C25	0.0884(3)	-0.0060(6)	-0.04079(16)	0.0523(16)*

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$.

Table 3. Selected Interatomic Distances (Å)

Atom1	Atom2	Distance	Atom1	Atom2	Distance
Cl	C1	1.763(4)	N3	C18	1.440(5)
O1	C4	1.232(5)	C1	C2	1.425(6)
O1	H3N	1.94†	C2	C3	1.415(6)
O2	C5	1.211(5)	C3	C4	1.425(6)
O3	C8	1.368(5)	C3	C11	1.409(6)
O3	C12	1.399(5)	C4	C5	1.561(6)
O4	C9	1.370(5)	C6	C7	1.370(6)
O4	C13	1.443(5)	C6	C11	1.425(5)
O5	C15	1.183(5)	C7	C8	1.398(6)
O6	C15	1.319(5)	C8	C9	1.394(6)
O6	C16	1.468(6)	C9	C10	1.405(6)
O7	C20	1.376(5)	C10	C11	1.408(6)
O7	C24	1.436(5)	C14	C15	1.516(6)
O8	C21	1.368(5)	C16	C17	1.316(7)
O8	C25	1.442(5)	C18	C19	1.403(5)
N1	C1	1.288(5)	C18	C23	1.369(6)
N1	C10	1.389(5)	C19	C20	1.378(6)
N2	C5	1.371(6)	C20	C21	1.404(6)
N2	C6	1.413(5)	C21	C22	1.387(6)
N2	C14	1.455(5)	C22	C23	1.382(6)
N3	C2	1.363(5)			

†Nonbonded distance.

Table 4. Selected Interatomic Angles (deg)

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C8	O3	C12	121.6(4)	O3	C8	C7	113.9(5)
C9	O4	C13	117.7(4)	O3	C8	C9	125.2(5)
C15	O6	C16	115.0(4)	C7	C8	C9	120.7(5)
C20	O7	C24	117.2(4)	O4	C9	C8	118.8(5)
C21	O8	C25	117.5(4)	O4	C9	C10	123.1(5)
C1	N1	C10	118.2(4)	C8	C9	C10	117.3(5)
C5	N2	C6	124.2(4)	N1	C10	C9	116.9(4)
C5	N2	C14	116.0(4)	N1	C10	C11	120.0(4)
C6	N2	C14	119.7(4)	C9	C10	C11	123.0(4)
C2	N3	C18	128.7(4)	C3	C11	C6	122.5(5)
Cl	C1	N1	113.2(4)	C3	C11	C10	119.9(4)
Cl	C1	C2	119.2(4)	C6	C11	C10	117.6(5)
N1	C1	C2	127.4(4)	N2	C14	C15	112.0(4)
N3	C2	C1	125.8(5)	O5	C15	O6	124.6(5)
N3	C2	C3	119.7(5)	O5	C15	C14	125.6(5)
C1	C2	C3	114.2(5)	O6	C15	C14	109.8(4)
C2	C3	C4	120.7(5)	O6	C16	C17	112.5(6)
C2	C3	C11	119.6(5)	N3	C18	C19	117.6(4)
C4	C3	C11	119.7(4)	N3	C18	C23	121.1(4)
O1	C4	C3	126.9(5)	C19	C18	C23	121.3(4)
O1	C4	C5	114.8(5)	C18	C19	C20	118.7(4)
C3	C4	C5	118.2(5)	O7	C20	C19	124.5(4)
O2	C5	N2	123.6(5)	O7	C20	C21	115.0(4)
O2	C5	C4	119.6(5)	C19	C20	C21	120.6(5)
N2	C5	C4	116.8(5)	O8	C21	C20	115.6(4)
N2	C6	C7	122.1(4)	O8	C21	C22	125.2(5)
N2	C6	C11	118.4(5)	C20	C21	C22	119.2(4)
C7	C6	C11	119.5(5)	C21	C22	C23	120.7(4)
C6	C7	C8	121.9(5)	C18	C23	C22	119.6(4)

Table 5. Torsional Angles (deg)

Atom1	Atom2	Atom3	Atom4	Angle	Atom1	Atom2	Atom3	Atom4	Angle
C12	O3	C8	C7	143.7(5)	C2	C3	C11	C10	4.8(6)
C12	O3	C8	C9	-41.1(7)	C4	C3	C11	C6	1.7(6)
C13	O4	C9	C8	126.2(5)	C4	C3	C11	C10	-176.5(4)
C13	O4	C9	C10	-64.7(6)	O1	C4	C5	O2	0.6(7)
C16	O6	C15	O5	1.1(9)	O1	C4	C5	N2	179.0(4)
C16	O6	C15	C14	-177.2(5)	C3	C4	C5	O2	177.6(4)
C15	O6	C16	C17	-149.2(7)	C3	C4	C5	N2	-4.0(6)
C24	O7	C20	C19	8.5(7)	N2	C6	C7	C8	-177.5(4)
C24	O7	C20	C21	-172.2(4)	C11	C6	C7	C8	2.9(7)
C25	O8	C21	C20	177.1(4)	N2	C6	C11	C3	0.1(6)
C25	O8	C21	C22	-1.5(7)	N2	C6	C11	C10	178.4(4)
C10	N1	C1	Cl	175.1(3)	C7	C6	C11	C3	179.7(4)
C10	N1	C1	C2	-1.3(7)	C7	C6	C11	C10	-2.0(6)
C1	N1	C10	C9	179.4(4)	C6	C7	C8	O3	173.6(4)
C1	N1	C10	C11	-4.0(6)	C6	C7	C8	C9	-1.9(7)
C6	N2	C5	O2	-175.5(5)	O3	C8	C9	O4	-5.3(7)
C6	N2	C5	C4	6.2(6)	O3	C8	C9	C10	-174.9(4)
C14	N2	C5	O2	1.1(7)	C7	C8	C9	O4	169.7(4)
C14	N2	C5	C4	-177.1(4)	C7	C8	C9	C10	0.1(7)
C5	N2	C6	C7	176.0(4)	O4	C9	C10	N1	8.1(6)
C5	N2	C6	C11	-4.4(6)	O4	C9	C10	C11	-168.4(4)
C14	N2	C6	C7	-0.5(6)	C8	C9	C10	N1	177.3(4)
C14	N2	C6	C11	179.1(4)	C8	C9	C10	C11	0.7(6)
C5	N2	C14	C15	-97.9(5)	N1	C10	C11	C3	2.1(6)
C6	N2	C14	C15	78.9(5)	N1	C10	C11	C6	-176.2(4)
C18	N3	C2	C1	24.8(7)	C9	C10	C11	C3	178.6(4)
C18	N3	C2	C3	-161.9(4)	C9	C10	C11	C6	0.3(6)
C2	N3	C18	C19	-123.9(5)	N2	C14	C15	O5	1.4(7)
C2	N3	C18	C23	58.3(7)	N2	C14	C15	O6	179.7(4)
Cl	C1	C2	N3	5.2(6)	N3	C18	C19	C20	-180.0(4)
Cl	C1	C2	C3	-168.3(3)	C23	C18	C19	C20	-2.1(7)
N1	C1	C2	N3	-178.5(4)	N3	C18	C23	C22	179.8(4)
N1	C1	C2	C3	7.9(7)	C19	C18	C23	C22	2.0(7)
N3	C2	C3	C4	-1.8(7)	C18	C19	C20	O7	-179.1(4)
N3	C2	C3	C11	176.8(4)	C18	C19	C20	C21	1.6(7)
C1	C2	C3	C4	172.2(4)	O7	C20	C21	O8	0.9(6)
C1	C2	C3	C11	-9.2(6)	O7	C20	C21	C22	179.6(4)
C2	C3	C4	O1	-4.5(7)	C19	C20	C21	O8	-179.8(4)
C2	C3	C4	C5	178.9(4)	C19	C20	C21	C22	-1.1(7)
C11	C3	C4	O1	176.9(5)	O8	C21	C22	C23	179.5(5)
C11	C3	C4	C5	0.3(6)	C20	C21	C22	C23	0.9(7)
C2	C3	C11	C6	-176.9(4)	C21	C22	C23	C18	-1.4(7)

Table 6. Anisotropic Displacement Parameters (U_{ij}, Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl	0.0448(8)	0.0512(7)	0.0215(7)	0.0128(7)	-0.0014(7)	-0.0090(8)
O1	0.024(2)	0.065(2)	0.032(2)	0.006(2)	0.0009(19)	-0.0007(19)
O2	0.032(3)	0.072(3)	0.031(2)	0.008(2)	0.0053(18)	-0.003(2)
O3	0.030(2)	0.075(3)	0.032(2)	0.003(2)	-0.0079(19)	0.010(2)
O4	0.032(2)	0.0416(19)	0.029(2)	-0.0046(18)	0.0024(18)	-0.0040(18)
O5	0.123(4)	0.034(2)	0.039(3)	-0.003(2)	0.023(2)	-0.010(3)
O6	0.134(4)	0.044(2)	0.016(2)	-0.003(2)	-0.007(2)	0.013(2)
O7	0.071(3)	0.041(2)	0.024(2)	-0.0042(19)	-0.0071(19)	0.024(2)
O8	0.073(3)	0.047(2)	0.017(2)	-0.0027(19)	-0.0027(19)	0.013(2)
N1	0.024(3)	0.029(2)	0.022(2)	0.000(2)	0.000(2)	-0.005(2)
N2	0.034(3)	0.037(2)	0.016(3)	0.007(2)	0.002(2)	-0.003(2)
N3	0.023(3)	0.056(3)	0.023(3)	0.003(2)	0.006(2)	0.006(2)
C1	0.034(3)	0.025(3)	0.022(3)	0.001(2)	-0.001(3)	0.000(3)
C2	0.028(3)	0.030(3)	0.030(3)	-0.001(3)	-0.003(3)	0.007(3)
C3	0.032(3)	0.025(3)	0.018(3)	0.004(2)	0.002(3)	-0.001(3)
C4	0.027(3)	0.038(3)	0.024(3)	0.002(3)	0.002(3)	0.005(3)
C5	0.032(4)	0.035(3)	0.026(3)	-0.001(3)	0.001(3)	0.003(3)
C6	0.027(3)	0.027(3)	0.019(3)	-0.002(2)	0.003(3)	-0.006(3)
C7	0.031(3)	0.036(3)	0.023(3)	0.003(2)	0.002(3)	-0.005(3)
C8	0.027(3)	0.034(3)	0.027(3)	-0.001(3)	-0.008(3)	0.005(3)
C9	0.026(3)	0.033(3)	0.026(3)	-0.007(3)	0.002(3)	-0.004(3)
C10	0.025(3)	0.026(2)	0.018(3)	-0.006(2)	-0.006(3)	0.003(2)
C11	0.016(3)	0.027(3)	0.019(3)	-0.008(2)	0.000(2)	-0.002(2)
C12	0.040(4)	0.087(5)	0.056(4)	-0.022(4)	-0.016(3)	0.025(4)
C13	0.046(4)	0.075(4)	0.035(4)	-0.020(3)	0.003(3)	-0.007(3)
C14	0.035(3)	0.036(3)	0.020(3)	0.007(3)	0.003(3)	-0.006(2)
C15	0.033(4)	0.050(3)	0.018(3)	-0.003(3)	0.002(3)	0.002(3)
C16	0.288(12)	0.081(5)	0.019(4)	-0.029(4)	-0.040(6)	0.070(6)
C17	0.201(10)	0.092(6)	0.110(7)	-0.050(6)	-0.080(7)	0.015(6)
C18	0.025(3)	0.036(3)	0.016(3)	0.002(3)	-0.006(2)	0.003(2)
C19	0.031(3)	0.036(3)	0.019(3)	-0.002(3)	-0.001(2)	0.008(3)
C20	0.032(3)	0.036(3)	0.024(3)	0.001(3)	-0.001(2)	0.008(3)
C21	0.045(4)	0.038(3)	0.015(3)	0.001(3)	-0.003(3)	0.003(3)
C22	0.038(4)	0.031(3)	0.023(3)	-0.006(3)	-0.003(3)	0.010(3)
C23	0.039(4)	0.033(3)	0.031(4)	0.008(3)	-0.002(3)	0.012(3)
C24	0.067(5)	0.046(3)	0.031(4)	0.003(3)	-0.001(3)	0.024(3)
C25	0.073(5)	0.058(4)	0.027(3)	-0.016(3)	0.001(3)	0.006(3)

The form of the anisotropic displacement parameter is:

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

Table 7. Derived Atomic Coordinates and Displacement Parameters for Hydrogen Atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H3N	0.0723	0.0888	0.1905	0.041
H7	0.2785	-0.1233	0.3621	0.036
H12A	0.4725	-0.0866	0.3463	0.073
H12B	0.4551	-0.0074	0.2980	0.073
H12C	0.4387	-0.2094	0.3098	0.073
H13A	0.4472	0.1032	0.1999	0.062
H13B	0.3775	0.0573	0.1845	0.062
H13C	0.4176	-0.0865	0.2115	0.062
H14A	0.1996	-0.2564	0.3828	0.036
H14B	0.1255	-0.2332	0.3861	0.036
H16A	0.2019	0.1498	0.4792	0.155
H16B	0.1371	0.0824	0.4998	0.155
H17A	0.2118	0.0757	0.5491	0.161
H17B	0.2513	-0.0545	0.5174	0.161
H17C	0.1864	-0.1195	0.5384	0.161
H19	0.0478	0.3237	0.1275	0.034
H22	0.1240	-0.1280	0.0344	0.037
H23	0.1399	-0.1431	0.1118	0.041
H24A	-0.0155	0.6146	0.0480	0.057
H24B	0.0444	0.5833	0.0792	0.057
H24C	-0.0208	0.4928	0.0920	0.057
H25A	0.0769	0.0247	-0.0717	0.063
H25B	0.0709	-0.1224	-0.0331	0.063
H25C	0.1340	-0.0103	-0.0382	0.063