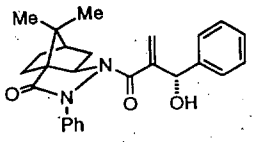
| | | | |
|---|---|---|---|
| C(2)-C(4)-H(4) | 107.5(6) | H(15')-C(15)-H(15") | 109.5(5) |
| C(5)-C(4)-H(4) | 107.2(6) | C(14)-C(16)-H(16) | 113.0(5) |
| C(4)-C(5)-C(6) | 115.3(6) | C(14)-C(16)-H(16') | 109.9(5) |
| C(4)-C(5)-H(5) | 108.7(5) | C(14)-C(16)-H(16") | 109.7(6) |
| C(4)-C(5)-H(5') | 108.9(6) | H(16)-C(16)-H(16') | 108.1(6) |
| C(6)-C(5)-H(5) | 107.3(6) | H(16)-C(16)-H(16") | 108.7(6) |
| C(6)-C(5)-H(5') | 106.2(6) | H(16')-C(16)-H(16") | 107.2(5) |
| H(5)-C(5)-H(5') | 110.5(6) | C(6)-C(1A)-C(2A) | 121.9(7) |
| C(5)-C(6)-C(1A) | 113.3(6) | C(6)-C(1A)-C(6A) | 121.0(8) |
| C(5)-C(6)-H(6) | 108.2(6) | C(2A)-C(1A)-C(6A) | 117.0(8) |
| C(5)-C(6)-H(6') | 109.5(6) | C(1A)-C(2A)-C(3A) | 120.4(7) |
| C(1A)-C(6)-H(6) | 109.1(6) | C(1A)-C(2A)-H(2A) | 119.6(9) |
| C(1A)-C(6)-H(6') | 110.6(7) | C(3A)-C(2A)-H(2A) | 120.0(10) |
| H(6)-C(6)-H(6') | 105.8(6) | C(2A)-C(3A)-C(4A) | 122.0(8) |
| O(3)-C(7)-N(2) | 122.3(5) | C(2A)-C(3A)-H(3A) | 117.1(9) |
| O(3)-C(7)-C(8) | 129.6(5) | C(4A)-C(3A)-H(3A) | 120.8(10) |
| N(2)-C(7)-C(8) | 108.1(5) | C(3A)-C(4A)-C(5A) | 117.4(8) |
| C(7)-C(8)-C(9) | 104.2(4) | C(3A)-C(4A)-H(4A) | 124.1(10) |
| C(7)-C(8)-C(13) | 120.9(5) | C(5A)-C(4A)-H(4A) | 118.4(10) |
| C(7)-C(8)-C(14) | 115.2(5) | C(4A)-C(5A)-C(6A) | 121.9(8) |
| C(9)-C(8)-C(13) | 106.9(5) | C(4A)-C(5A)-H(5A) | 118.2(10) |
| C(9)-C(8)-C(14) | 104.5(5) | C(6A)-C(5A)-H(5A) | 119.7(10) |
| C(13)-C(8)-C(14) | 103.8(4) | C(1A)-C(6A)-C(5A) | 121.2(8) |
| N(1)-C(9)-C(8) | 103.1(5) | C(1A)-C(6A)-H(6A) | 120.4(8) |
| N(1)-C(9)-C(10) | 119.4(5) | C(5A)-C(6A)-H(6A) | 118.3(8) |
| N(1)-C(9)-H(9) | 110.5(4) | N(2)-C(1B)-C(2B) | 119.2(6) |
| C(8)-C(9)-C(10) | 102.7(4) | N(2)-C(1B)-C(6B) | 119.3(6) |
| C(8)-C(9)-H(9) | 110.5(5) | C(2B)-C(1B)-C(6B) | 121.4(5) |
| C(10)-C(9)-H(9) | 109.9(5) | C(1B)-C(2B)-C(3B) | 118.9(6) |
| C(9)-C(10)-C(11) | 101.7(5) | C(1B)-C(2B)-H(2B) | 120.6(6) |
| C(9)-C(10)-H(10) | 111.6(5) | C(3B)-C(2B)-H(2B) | 120.5(7) |
| C(9)-C(10)-H(10') | 111.7(5) | C(2B)-C(3B)-C(4B) | 120.1(7) |
| C(11)-C(10)-H(10) | 112.3(5) | C(2B)-C(3B)-H(3B) | 120.6(9) |
| C(11)-C(10)-H(10') | 113.6(6) | C(4B)-C(3B)-H(3B) | 119.3(8) |
| H(10)-C(10)-H(10') | 106.1(6) | C(3B)-C(4B)-C(5B) | 120.1(6) |
| C(10)-C(11)-C(12) | 109.7(5) | C(3B)-C(4B)-H(4B) | 118.7(10) |
| C(10)-C(11)-C(14) | 102.1(5) | C(5B)-C(4B)-H(4B) | 121.2(9) |
| C(10)-C(11)-H(11) | 114.4(6) | C(4B)-C(5B)-C(6B) | 120.3(7) |
| C(12)-C(11)-C(14) | 103.0(6) | C(4B)-C(5B)-H(5B) | 120.7(7) |
| C(12)-C(11)-H(11) | 112.7(5) | C(6B)-C(5B)-H(5B) | 119.0(8) |
| C(14)-C(11)-H(11) | 113.9(6) | C(1B)-C(6B)-C(5B) | 119.2(7) |
| C(11)-C(12)-C(13) | 103.2(4) | C(1B)-C(6B)-H(6B) | 117.1(5) |
| C(11)-C(12)-H(12) | 113.0(5) | C(5B)-C(6B)-H(6B) | 123.5(7) |
| C(11)-C(12)-H(12') | 113.6(7) | | |

CH1140

Space Group and Cell Dimensions Monoclinic, C 2
a 26.526(4) b 9.0300(20) c 12.0787(22)
beta 116.763(15)
Volume 2583.3(8)A**3




Empirical formula : C27 H30 Cl2 N2 O3

Cell dimensions were obtained from 25 reflections with 2Theta angle
in the range 18.40 - 26.38 degrees.

Crystal dimensions : 0.45 X 0.30 X 0.25 mm

FW = 501.45 Z = 4 F(000) = 1055.86

Dcalc 1.289Mg.m-3, mu 0.28mm-1, lambda 0.70930A, 2Theta(max) 51.8

The intensity data were collected on a Nonius diffractometer,
using the theta/2theta scan mode.
The h,k,l ranges are :-- -32 29, 0 11, 0 14
No. of reflections measured 2851
No. of unique reflections 2718
No. of reflections with Inet > 2.0sigma(Inet) 1796
Absorption corrections were made.
The minimum and maximum transmission factors are 0.886843 and 0.999563.

The last least squares cycle was calculated with
64 atoms, 307 parameters and 1796 out of 2718 reflections.
Unit weights were used.

The residuals are as follows :--
For significant reflections, RF 0.080, Rw 0.076 GoF 2.21
For all reflections, RF 0.115, Rw 0.093.
where RF = Sum(Fo-Fc)/Sum(Fo),
Rw = Sqrt[Sum(w(Fo-Fc)**2)/Sum(wFo**2)] and
GoF = Sqrt[Sum(w(Fo-Fc)**2)/(No. of reflns - No. of params.)]
The maximum shift/sigma ratio was 0.001.

In the last D-map, the deepest hole was -0.570e/A**3,
and the highest peak 0.720e/A**3.

Secondary ext. coeff. = 0.289482 sigma = 0.084338

CH1140

The following Atoms are from the CD File

* Indicates that there are Symmetry Equivalents of an atom.

| Name | x | y | z |
|---|---|---|---|
| O(1) | -0.27321 | -0.84909 | -0.09292 |
| O(2) | -0.23305 | -1.20409 | -0.34450 |
| O(3) | -0.26804 | -1.64253 | -0.43040 |
| N(1) | -0.25068 | -1.05196 | -0.17409 |
| N(2) | -0.27191 | -1.19826 | -0.21263 |
| C(1) | -0.28583 | -0.97021 | -0.14241 |
| C(2) | -0.25798 | -1.26487 | -0.29378 |
| C(3) | -0.27058 | -1.42918 | -0.31115 |
| C(4) | -0.26408 | -1.51404 | -0.21764 |
| C(5) | -0.28488 | -1.48801 | -0.44082 |
| C(6) | -0.32115 | -1.21820 | -0.18650 |
| C(7) | -0.33875 | -1.05535 | -0.17839 |
| C(8) | -0.36806 | -1.06053 | -0.09530 |
| C(9) | -0.42435 | -1.14227 | -0.18342 |
| C(10) | -0.41982 | -1.16186 | -0.30615 |
| C(11) | -0.37622 | -1.28546 | -0.28701 |
| C(12) | -0.38863 | -1.02301 | -0.31069 |
| C(13) | -0.37133 | -1.01778 | -0.41647 |
| C(14) | -0.41884 | -0.87762 | -0.31551 |
| C(1A) | -0.19077 | -1.03109 | -0.11696 |
| C(2A) | -0.15740 | -1.11991 | -0.01858 |
| C(3A) | -0.09926 | -1.09816 | 0.03964 |
| C(4A) | -0.07603 | -0.98612 | 0.00085 |
| C(5A) | -0.11004 | -0.89595 | -0.09409 |
| C(6A) | -0.16903 | -0.91849 | -0.15548 |
| C(1B) | -0.34869 | -1.47502 | -0.52044 |
| C(2B) | -0.38541 | -1.57591 | -0.51160 |
| C(3B) | -0.44267 | -1.55560 | -0.57333 |
| C(4B) | -0.46454 | -1.43536 | -0.64615 |
| C(5B) | -0.42852 | -1.33093 | -0.66243 |
| C(6B) | -0.37114 | -1.35342 | -0.59686 |
| Cl(1) | -0.03449 | -0.33352 | -0.18960 |
| Cl(2) | -0.12634 | -0.48479 | -0.23132 |
| C(15) | -0.09861 | -0.32759 | -0.22604 |
| HO(3) | -0.29394 | -1.67950 | -0.40011 |
| H(4) | -0.25308 | -1.46867 | -0.13661 |
| H(4') | -0.27049 | -1.62730 | -0.22695 |
| H(5) | -0.26249 | -1.42656 | -0.47941 |
| H(6) | -0.30907 | -1.27909 | -0.09692 |
| H(8) | -0.37527 | -0.95725 | -0.06303 |
| H(8') | -0.34267 | -1.12980 | -0.00643 |
| H(9) | -0.42679 | -1.25723 | -0.14855 |
| H(9') | -0.46069 | -1.08482 | -0.19897 |
| H(10) | -0.46038 | -1.18288 | -0.38740 |
| H(11) | -0.38735 | -1.39511 | -0.25755 |
| H(11') | -0.37209 | -1.31409 | -0.37189 |
| H(13) | -0.40167 | -1.06872 | -0.50858 |
| H(13') | -0.36176 | -0.91073 | -0.43639 |
| H(13") | -0.33168 | -1.08854 | -0.39364 |
| H(14) | -0.43597 | -0.89865 | -0.25062 |
| H(14') | -0.38898 | -0.79193 | -0.28729 |
| H(14") | -0.45219 | -0.86003 | -0.40781 |
| H(2A) | -0.17712 | -1.19469 | 0.01664 |

| | | | |
|---|---|---|---|
| H(4A) | -0.03541 | -0.97293 | 0.04047 |
| H(5A) | -0.09074 | -0.81784 | -0.12199 |
| H(6A) | -0.19348 | -0.85982 | -0.22598 |
| H(2B) | -0.37190 | -1.67117 | -0.46106 |
| H(3B) | -0.46837 | -1.62358 | -0.55918 |
| H(4B) | -0.50552 | -1.44192 | -0.69848 |
| H(5B) | -0.44749 | -1.25632 | -0.71846 |
| H(6B) | -0.34468 | -1.28162 | -0.60444 |
| H(15) | -0.11335 | -0.24294 | -0.18031 |
| H(15') | -0.12910 | -0.27658 | -0.33635 |

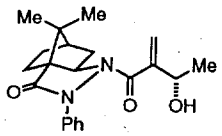
CH1140

| | | | |
|---|---|---|---|
| O(1)-C(1) | 1.219(17) | C(12)-C(14) | 1.525(18) |
| O(2)-C(2) | 1.216(13) | C(13)-H(13) | 1.136(10) |
| O(3)-C(5) | 1.453(17) | C(13)-H(13') | 1.054(14) |
| O(3)-HO(3) | 0.971(8) | C(13)-H(13") | 1.153(11) |
| N(1)-N(2) | 1.429(15) | C(14)-H(14) | 1.085(12) |
| N(1)-C(1) | 1.372(14) | C(14)-H(14') | 1.048(14) |
| N(1)-C(1A) | 1.432(11) | C(14)-H(14") | 1.078(11) |
| N(2)-C(2) | 1.336(13) | C(1A)-C(2A) | 1.375(16) |
| N(2)-C(6) | 1.486(11) | C(1A)-C(6A) | 1.351(18) |
| C(1)-C(7) | 1.484(14) | C(2A)-C(3A) | 1.391(15) |
| C(2)-C(3) | 1.515(19) | C(2A)-H(2A) | 1.054(12) |
| C(3)-C(4) | 1.311(15) | C(3A)-C(4A) | 1.372(21) |
| C(3)-C(5) | 1.531(13) | C(3A)-H(3A) | 1.008(13) |
| C(4)-H(4) | 0.977(10) | C(4A)-C(5A) | 1.364(22) |
| C(4)-H(4') | 1.035(13) | C(4A)-H(4A) | 0.969(10) |
| C(5)-C(1B) | 1.529(13) | C(5A)-C(6A) | 1.412(17) |
| C(5)-H(5) | 1.062(10) | C(5A)-H(5A) | 1.014(13) |
| C(6)-C(7) | 1.559(17) | C(6A)-H(6A) | 0.964(13) |
| C(6)-C(11) | 1.543(13) | C(1B)-C(2B) | 1.372(19) |
| C(6)-H(6) | 1.124(9) | C(1B)-C(6B) | 1.385(18) |
| C(7)-C(8) | 1.522(12) | C(2B)-C(3B) | 1.370(18) |
| C(7)-C(12) | 1.576(12) | C(2B)-H(2B) | 1.022(15) |
| C(8)-C(9) | 1.573(15) | C(3B)-C(4B) | 1.35(3) |
| C(8)-H(8) | 1.061(12) | C(3B)-H(3B) | 0.988(16) |
| C(8)-H(8') | 1.160(11) | C(4B)-C(5B) | 1.42(3) |
| C(9)-C(10) | 1.550(14) | C(4B)-H(4B) | 0.983(13) |
| C(9)-H(9) | 1.133(14) | C(5B)-C(6B) | 1.378(16) |
| C(9)-H(9') | 1.035(10) | C(5B)-H(5B) | 0.926(14) |
| C(10)-C(11) | 1.548(16) | C(6B)-H(6B) | 0.990(13) |
| C(10)-C(12) | 1.517(18) | Cl(1)-C(15) | 1.554(17) |
| C(10)-H(10) | 1.098(9) | Cl(2)-C(15) | 1.587(24) |
| C(11)-H(11) | 1.135(12) | C(15)-H(15) | 1.112(21) |
| C(11)-H(11') | 1.110(9) | C(15)-H(15') | 1.293(23) |
| C(12)-C(13) | 1.541(13) | | |

| | | | |
|---|---|---|---|
| C(5)-O(3)-HO(3) | 96.9(7) | C(7)-C(12)-C(10) | 91.9(8) |
| N(2)-N(1)-C(1) | 111.8(8) | C(7)-C(12)-C(13) | 114.8(7) |
| N(2)-N(1)-C(1A) | 117.9(9) | C(7)-C(12)-C(14) | 112.6(9) |
| C(1)-N(1)-C(1A) | 122.8(9) | C(10)-C(12)-C(13) | 115.2(10) |
| N(1)-N(2)-C(2) | 117.2(8) | C(10)-C(12)-C(14) | 115.3(9) |
| N(1)-N(2)-C(6) | 107.3(8) | C(13)-C(12)-C(14) | 106.8(10) |
| C(2)-N(2)-C(6) | 131.4(9) | C(12)-C(13)-H(13) | 117.1(9) |
| O(1)-C(1)-N(1) | 123.3(9) | C(12)-C(13)-H(13') | 114.2(11) |
| O(1)-C(1)-C(7) | 128.6(9) | C(12)-C(13)-H(13") | 111.4(8) |
| N(1)-C(1)-C(7) | 108.2(10) | H(13)-C(13)-H(13') | 106.6(8) |
| O(2)-C(2)-N(2) | 124.0(12) | H(13)-C(13)-H(13") | 100.2(9) |
| O(2)-C(2)-C(3) | 120.6(10) | H(13')-C(13)-H(13") | 106.0(9) |
| N(2)-C(2)-C(3) | 115.2(9) | C(12)-C(14)-H(14) | 101.9(10) |
| C(2)-C(3)-C(4) | 121.2(9) | C(12)-C(14)-H(14') | 108.4(10) |
| C(2)-C(3)-C(5) | 114.8(9) | C(12)-C(14)-H(14") | 109.8(11) |
| C(4)-C(3)-C(5) | 123.7(11) | H(14)-C(14)-H(14') | 113.3(12) |
| C(3)-C(4)-H(4) | 118.8(12) | H(14)-C(14)-H(14") | 110.9(10) |
| C(3)-C(4)-H(4') | 122.2(10) | H(14')-C(14)-H(14") | 101.3(14) |

| | |
|---|---|
| O(3)-C(5)-H(5) | 109.2(8) |
| C(3)-C(5)-C(1B) | 108.1(7) |
| C(3)-C(5)-H(5) | 109.7(9) |
| C(1B)-C(5)-H(5) | 111.3(9) |
| N(2)-C(6)-C(7) | 102.5(9) |
| N(2)-C(6)-C(11) | 119.5(7) |
| N(2)-C(6)-H(6) | 111.5(7) |
| C(7)-C(6)-C(11) | 102.5(8) |
| C(7)-C(6)-H(6) | 111.4(8) |
| C(11)-C(6)-H(6) | 108.9(9) |
| C(1)-C(7)-C(6) | 103.7(8) |
| C(1)-C(7)-C(8) | 121.3(8) |
| C(1)-C(7)-C(12) | 116.9(9) |
| C(6)-C(7)-C(8) | 105.6(9) |
| C(6)-C(7)-C(12) | 104.4(8) |
| C(8)-C(7)-C(12) | 103.3(7) |
| C(7)-C(8)-C(9) | 100.3(7) |
| C(7)-C(8)-H(8) | 116.4(11) |
| C(7)-C(8)-H(8') | 111.8(8) |
| C(9)-C(8)-H(8) | 112.8(8) |
| C(9)-C(8)-H(8') | 110.6(10) |
| H(8)-C(8)-H(8') | 105.0(7) |
| C(8)-C(9)-C(10) | 104.0(7) |
| C(8)-C(9)-H(9) | 112.0(8) |
| C(8)-C(9)-H(9') | 114.2(11) |
| C(10)-C(9)-H(9) | 107.0(10) |
| C(10)-C(9)-H(9') | 110.5(8) |
| H(9)-C(9)-H(9') | 108.8(9) |
| C(9)-C(10)-C(11) | 108.3(9) |
| C(9)-C(10)-C(12) | 102.6(9) |
| C(9)-C(10)-H(10) | 114.1(8) |
| C(11)-C(10)-C(12) | 102.6(8) |
| C(11)-C(10)-H(10) | 112.0(10) |
| C(12)-C(10)-H(10) | 116.2(10) |
| C(6)-C(11)-C(10) | 102.3(9) |
| C(6)-C(11)-H(11) | 112.6(8) |
| C(6)-C(11)-H(11') | 111.5(7) |
| C(10)-C(11)-H(11) | 113.2(8) |
| C(10)-C(11)-H(11') | 114.1(9) |
| H(11)-C(11)-H(11') | 103.5(10) |
| C(1A)-C(2A)-C(3A) | 119.3(11) |
| C(1A)-C(2A)-H(2A) | 118.6(9) |
| C(3A)-C(2A)-H(2A) | 121.7(11) |
| C(2A)-C(3A)-C(4A) | 119.9(12) |
| C(2A)-C(3A)-H(3A) | 117.4(14) |
| C(4A)-C(3A)-H(3A) | 122.2(11) |
| C(3A)-C(4A)-C(5A) | 119.9(10) |
| C(3A)-C(4A)-H(4A) | 119.5(14) |
| C(5A)-C(4A)-H(4A) | 120.6(14) |
| C(4A)-C(5A)-C(6A) | 120.8(12) |
| C(4A)-C(5A)-H(5A) | 116.9(13) |
| C(6A)-C(5A)-H(5A) | 122.1(15) |
| C(1A)-C(6A)-C(5A) | 118.1(12) |
| C(1A)-C(6A)-H(6A) | 119.8(11) |
| C(5A)-C(6A)-H(6A) | 122.0(13) |
| C(5)-C(1B)-C(2B) | 121.6(11) |
| C(5)-C(1B)-C(6B) | 120.1(11) |
| C(2B)-C(1B)-C(6B) | 118.1(10) |
| C(1B)-C(2B)-C(3B) | 121.4(14) |
| C(1B)-C(2B)-H(2B) | 122.4(11) |
| C(3B)-C(2B)-H(2B) | 116.3(14) |
| C(2B)-C(3B)-C(4B) | 120.5(15) |
| C(2B)-C(3B)-H(3B) | 120.3(17) |
| C(4B)-C(3B)-H(3B) | 119.0(14) |
| C(3B)-C(4B)-C(5B) | 120.4(12) |
| C(3B)-C(4B)-H(4B) | 113.3(19) |
| C(5B)-C(4B)-H(4B) | 124.8(17) |
| C(4B)-C(5B)-C(6B) | 117.5(13) |
| C(4B)-C(5B)-H(5B) | 114.0(13) |
| C(6B)-C(5B)-H(5B) | 128.5(17) |
| C(1B)-C(6B)-C(5B) | 122.1(13) |
| C(1B)-C(6B)-H(6B) | 118.1(10) |
| C(5B)-C(6B)-H(6B) | 119.7(14) |
| Cl(1)-C(15)-Cl(2) | 114.1(13) |
| Cl(1)-C(15)-H(15) | 117.9(16) |
| Cl(1)-C(15)-H(15') | 113.6(15) |
| Cl(2)-C(15)-H(15) | 112.0(14) |
| Cl(2)-C(15)-H(15') | 102.3(14) |
| H(15)-C(15)-H(15') | 94.0(14) |

CH1132




Space Group and Cell Dimensions Orthorhombic P 212121
a 8.870(3) b 11.947(3) c 20.158(4)
Volume 2136.2(10)A**3

Empirical formula : C22 H30 N2 O4

Cell dimensions were obtained from 25 reflections with 2Theta angle in the range 18.98 - 29.12 degrees.

Crystal dimensions : 0.35 X 0.30 X 0.25 mm

FW = 386.49 Z = 4 F(000) = 831.87

Dcalc 1.202Mg.m-3, mu 0.08mm-1, lambda 0.70930A, 2Theta(max) 55.8

The intensity data were collected on a Nonius diffractometer, using the theta/2theta scan mode.
The h,k,l ranges are :-- 0 11, 0 15, 0 26
No. of reflections measured 2915
No. of unique reflections 2915
No. of reflections with Inet > 2.0sigma(Inet) 1539
Absorption corrections were made.
The minimum and maximum transmission factors are 0.908341 and 0.999300.

The last least squares cycle was calculated with
58 atoms, 224 parameters and 1539 out of 2915 reflections.
Unit weights were used.

The residuals are as follows :--
For significant reflections, RF 0.078, Rw 0.080 GoF 1.84
For all reflections, RF 0.146, Rw 0.120.
where RF = Sum(Fo-Fc)/Sum(Fo),
Rw = Sqrt[Sum(w(Fo-Fc)**2)/Sum(wFo**2)] and
GoF = Sqrt[Sum(w(Fo-Fc)**2)/(No. of reflns - No. of params.)]
The maximum shift/sigma ratio was 0.001.

In the last D-map, the deepest hole was -0.330e/A**3,
and the highest peak 0.260e/A**3.

Secondary ext. coeff. = 0.273179 sigma = 0.045565

CH1132

The following Atoms are from the CD File

* Indicates that there are Symmetry Equivalents of an atom.

| Name | x | y | z |
|---|---|---|---|
| O(1) | 0.29809 | 0.60235 | 0.12320 |
| O(2) | 0.73748 | 0.59758 | 0.03944 |
| O(3) | 1.10893 | 0.29402 | 0.03885 |
| N(1) | 0.75155 | 0.41132 | 0.06106 |
| N(2) | 0.90217 | 0.40793 | 0.03219 |
| C(1) | 0.53254 | 0.63589 | 0.17701 |
| C(2) | 0.45622 | 0.61174 | 0.11043 |
| C(3) | 0.51213 | 0.50754 | 0.07972 |
| C(4) | 0.42933 | 0.42317 | 0.06192 |
| C(5) | 0.68123 | 0.51008 | 0.05948 |
| C(6) | 0.73329 | 0.31272 | 0.10365 |
| C(7) | 0.66525 | 0.32684 | 0.17366 |
| C(8) | 0.79098 | 0.27664 | 0.21841 |
| C(9) | 0.80795 | 0.15190 | 0.20130 |
| C(10) | 0.89613 | 0.15167 | 0.13297 |
| C(11) | 0.90288 | 0.27569 | 0.11816 |
| C(12) | 0.98623 | 0.32178 | 0.06140 |
| C(13) | 0.93487 | 0.32989 | 0.18649 |
| C(14) | 0.94386 | 0.45663 | 0.18942 |
| C(15) | 1.08728 | 0.28562 | 0.21679 |
| C(1A) | 0.92903 | 0.45454 | -0.03090 |
| C(2A) | 0.83810 | 0.42390 | -0.08327 |
| C(3A) | 0.86908 | 0.47255 | -0.14594 |
| C(4A) | 0.98531 | 0.54444 | -0.15366 |
| C(5A) | 1.07421 | 0.57136 | -0.10075 |
| C(6A) | 1.04964 | 0.52560 | -0.03883 |
| O(4) | 0.15420 | 0.78185 | 0.07051 |
| C(16) | 0.00456 | 0.79647 | 0.08229 |
| HO(1) | 0.26134 | 0.64855 | 0.08664 |
| H(1) | 0.64973 | 0.66146 | 0.16186 |
| H(1') | 0.54258 | 0.56006 | 0.20647 |
| H(1") | 0.48545 | 0.70145 | 0.20700 |
| H(2) | 0.48142 | 0.68781 | 0.07928 |
| H(4) | 0.46932 | 0.36908 | 0.02763 |
| H(4') | 0.32756 | 0.39974 | 0.08218 |
| H(6) | 0.67175 | 0.24770 | 0.07779 |
| H(7) | 0.63968 | 0.41415 | 0.18597 |
| H(7') | 0.55791 | 0.28000 | 0.17976 |
| H(8) | 0.77797 | 0.29394 | 0.27239 |
| H(9) | 0.86872 | 0.10681 | 0.23982 |
| H(9') | 0.69679 | 0.11361 | 0.19607 |
| H(10) | 0.83912 | 0.10432 | 0.09284 |
| H(10') | 1.01107 | 0.11361 | 0.13640 |
| H(14) | 0.93000 | 0.47720 | 0.24110 |
| H(14') | 0.85262 | 0.49595 | 0.16006 |
| H(14") | 1.04993 | 0.49118 | 0.17041 |
| H(15) | 1.07190 | 0.19281 | 0.21953 |
| H(15') | 1.10991 | 0.31681 | 0.26518 |
| H(15") | 1.18159 | 0.30185 | 0.18406 |
| H(2A) | 0.75151 | 0.36936 | -0.07560 |
| H(3A) | 0.80152 | 0.44898 | -0.18355 |

| | | | |
|---|---|---|---|
| HO(4) | 0.16317 | 0.71961 | 0.03507 |
| H(16) | -0.04461 | 0.83716 | 0.04425 |
| H(16') | 0.00741 | 0.85740 | 0.12847 |
| H(16") | -0.05097 | 0.72396 | 0.10127 |

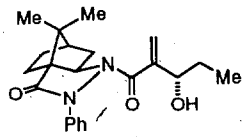
CH1132

| | | | |
|---|---|---|---|
| O(1)-C(2) | 1.431(11) | C(9)-H(9') | 1.092(10) |
| O(1)-HO(1) | 0.977(9) | C(10)-C(11) | 1.513(12) |
| O(2)-C(5) | 1.227(11) | C(10)-H(10) | 1.109(10) |
| O(3)-C(12) | 1.225(9) | C(10)-H(10') | 1.119(9) |
| N(1)-N(2) | 1.458(8) | C(11)-C(12) | 1.469(11) |
| N(1)-C(5) | 1.335(11) | C(11)-C(13) | 1.548(11) |
| N(1)-C(6) | 1.467(10) | C(13)-C(14) | 1.517(14) |
| N(2)-C(12) | 1.401(11) | C(13)-C(15) | 1.575(12) |
| N(2)-C(1A) | 1.409(9) | C(14)-H(14) | 1.078(9) |
| C(1)-C(2) | 1.531(17) | C(14)-H(14') | 1.107(10) |
| C(1)-H(1) | 1.126(12) | C(14)-H(14") | 1.097(10) |
| C(1)-H(1') | 1.087(12) | C(15)-H(15) | 1.119(13) |
| C(1)-H(1") | 1.074(10) | C(15)-H(15') | 1.063(10) |
| C(2)-C(3) | 1.476(15) | C(15)-H(15") | 1.083(10) |
| C(2)-H(2) | 1.127(10) | C(1A)-C(2A) | 1.378(12) |
| C(3)-C(4) | 1.298(15) | C(1A)-C(6A) | 1.375(12) |
| C(3)-C(5) | 1.555(11) | C(2A)-C(3A) | 1.417(14) |
| C(4)-H(4) | 1.011(11) | C(2A)-H(2A) | 1.019(9) |
| C(4)-H(4') | 1.030(9) | C(3A)-C(4A) | 1.351(15) |
| C(6)-C(7) | 1.544(11) | C(3A)-H(3A) | 1.007(11) |
| C(6)-C(11) | 1.595(10) | C(4A)-C(5A) | 1.365(14) |
| C(6)-H(6) | 1.083(8) | C(4A)-H(4A) | 1.017(10) |
| C(7)-C(8) | 1.555(12) | C(5A)-C(6A) | 1.380(13) |
| C(7)-H(7) | 1.096(9) | C(5A)-H(5A) | 1.070(10) |
| C(7)-H(7') | 1.111(8) | C(6A)-H(6A) | 1.032(9) |
| C(8)-C(9) | 1.537(15) | O(4)-C(16) | 1.360(16) |
| C(8)-C(13) | 1.565(12) | O(4)-HO(4) | 1.034(14) |
| C(8)-H(8) | 1.113(8) | C(16)-H(16) | 1.007(15) |
| C(9)-C(10) | 1.584(13) | C(16)-H(16') | 1.182(17) |
| C(9)-H(9) | 1.088(10) | C(16)-H(16") | 1.068(17) |

| | | | |
|---|---|---|---|
| C(2)-O(1)-HO(1) | 98.5(7) | C(11)-C(10)-H(10) | 111.9(7) |
| N(2)-N(1)-C(5) | 116.3(6) | C(11)-C(10)-H(10') | 112.0(8) |
| N(2)-N(1)-C(6) | 108.2(6) | H(10)-C(10)-H(10') | 104.7(7) |
| C(5)-N(1)-C(6) | 132.2(6) | C(6)-C(11)-C(10) | 105.7(6) |
| N(1)-N(2)-C(12) | 109.9(6) | C(6)-C(11)-C(12) | 103.2(6) |
| N(1)-N(2)-C(1A) | 120.3(6) | C(6)-C(11)-C(13) | 102.7(6) |
| C(12)-N(2)-C(1A) | 125.4(6) | C(10)-C(11)-C(12) | 122.7(7) |
| C(2)-C(1)-H(1) | 102.8(8) | C(10)-C(11)-C(13) | 104.0(7) |
| C(2)-C(1)-H(1') | 111.0(10) | C(12)-C(11)-C(13) | 116.3(7) |
| C(2)-C(1)-H(1") | 117.3(10) | O(3)-C(12)-N(2) | 121.1(7) |
| H(1)-C(1)-H(1') | 107.4(10) | O(3)-C(12)-C(11) | 129.4(8) |
| H(1)-C(1)-H(1") | 108.3(10) | N(2)-C(12)-C(11) | 109.6(7) |
| H(1')-C(1)-H(1") | 109.4(9) | C(8)-C(13)-C(11) | 92.7(6) |
| O(1)-C(2)-C(1) | 106.9(8) | C(8)-C(13)-C(14) | 115.6(8) |
| O(1)-C(2)-C(3) | 109.8(9) | C(8)-C(13)-C(15) | 113.8(7) |
| O(1)-C(2)-H(2) | 111.0(8) | C(11)-C(13)-C(14) | 117.5(7) |
| C(1)-C(2)-C(3) | 112.2(8) | C(11)-C(13)-C(15) | 111.2(8) |
| C(1)-C(2)-H(2) | 104.4(9) | C(14)-C(13)-C(15) | 106.0(8) |
| C(3)-C(2)-H(2) | 112.3(8) | C(13)-C(14)-H(14) | 105.0(8) |
| C(2)-C(3)-C(4) | 125.5(8) | C(13)-C(14)-H(14') | 111.4(7) |
| C(2)-C(3)-C(5) | 114.7(8) | C(13)-C(14)-H(14") | 114.0(9) |
| C(4)-C(3)-C(5) | 119.2(8) | H(14)-C(14)-H(14') | 109.7(9) |

| | | | |
|---|---|---|---|
| O(2)-C(5)-N(1) | 124.8(7) | C(13)-C(15)-H(15') | 113.6(9) |
| O(2)-C(5)-C(3) | 119.7(8) | C(13)-C(15)-H(15") | 111.5(8) |
| N(1)-C(5)-C(3) | 115.3(8) | H(15)-C(15)-H(15') | 109.0(8) |
| N(1)-C(6)-C(7) | 119.4(7) | H(15)-C(15)-H(15") | 107.6(10) |
| N(1)-C(6)-C(11) | 103.1(5) | H(15')-C(15)-H(15") | 110.5(9) |
| N(1)-C(6)-H(6) | 110.5(6) | N(2)-C(1A)-C(2A) | 119.2(7) |
| C(7)-C(6)-C(11) | 103.4(6) | N(2)-C(1A)-C(6A) | 118.7(7) |
| C(7)-C(6)-H(6) | 108.7(6) | C(2A)-C(1A)-C(6A) | 122.0(8) |
| C(11)-C(6)-H(6) | 111.4(6) | C(1A)-C(2A)-C(3A) | 117.4(8) |
| C(6)-C(7)-C(8) | 102.0(6) | C(1A)-C(2A)-H(2A) | 119.7(8) |
| C(6)-C(7)-H(7) | 113.1(7) | C(3A)-C(2A)-H(2A) | 122.9(9) |
| C(6)-C(7)-H(7') | 112.4(7) | C(2A)-C(3A)-C(4A) | 120.8(10) |
| C(8)-C(7)-H(7) | 112.6(7) | C(2A)-C(3A)-H(3A) | 116.2(10) |
| C(8)-C(7)-H(7') | 110.9(7) | C(4A)-C(3A)-H(3A) | 123.1(10) |
| H(7)-C(7)-H(7') | 106.1(7) | C(3A)-C(4A)-C(5A) | 120.0(9) |
| C(7)-C(8)-C(9) | 108.3(7) | C(3A)-C(4A)-H(4A) | 120.5(10) |
| C(7)-C(8)-C(13) | 100.9(6) | C(5A)-C(4A)-H(4A) | 119.0(10) |
| C(7)-C(8)-H(8) | 114.9(7) | C(4A)-C(5A)-C(6A) | 121.5(9) |
| C(9)-C(8)-C(13) | 102.8(7) | C(4A)-C(5A)-H(5A) | 118.6(9) |
| C(9)-C(8)-H(8) | 114.2(8) | C(6A)-C(5A)-H(5A) | 119.5(9) |
| C(13)-C(8)-H(8) | 114.3(7) | C(1A)-C(6A)-C(5A) | 118.2(8) |
| C(8)-C(9)-C(10) | 104.2(7) | C(1A)-C(6A)-H(6A) | 119.0(8) |
| C(8)-C(9)-H(9) | 111.6(9) | C(5A)-C(6A)-H(6A) | 121.1(9) |
| C(8)-C(9)-H(9') | 109.8(8) | C(16)-O(4)-HO(4) | 106.7(12) |
| C(10)-C(9)-H(9) | 112.0(8) | O(4)-C(16)-H(16) | 110.6(12) |
| C(10)-C(9)-H(9') | 111.2(9) | O(4)-C(16)-H(16') | 101.3(12) |
| H(9)-C(9)-H(9') | 108.0(8) | O(4)-C(16)-H(16") | 114.1(14) |
| C(9)-C(10)-C(11) | 100.9(7) | H(16)-C(16)-H(16') | 108.2(16) |
| C(9)-C(10)-H(10) | 114.2(8) | H(16)-C(16)-H(16") | 117.7(14) |
| C(9)-C(10)-H(10') | 113.4(8) | H(16')-C(16)-H(16") | 103.1(11) |

Table 1. Crystal data and structure refinement for IC6498.




| | |
|---|---|
| Identification code | ic6498 |
| Empirical formula | $C_{22}H_{28}N_2O_3$ |
| Formula weight | 368.46 |
| Temperature | 295(2) K |
| Wavelength | 0.71073 Å |
| Crystal system | Orthorhombic |
| Space group | $P2_12_12_1$ |
| Unit cell dimensions | $a$ = 9.3369(2) Å alpha = 90°<br>$b$ = 9.8804(3) Å beta = 90°<br>$c$ = 43.7475(14) Å gamma = 90° |
| Volume, Z | 4035.8(2) Å$^3$, 8 |
| Density (calculated) | 1.213 Mg/m$^3$ |
| Absorption coefficient | 0.081 mm$^{-1}$ |
| F(000) | 1584 |
| Crystal size | 0.4 x 0.3 x 0.25 mm |
| θ range for data collection | 0.93 to 23.25° |
| Limiting indices | $-10 \le h \le 9$, $-4 \le k \le 10$, $-25 \le l \le 48$ |
| Reflections collected | 10316 |
| Independent reflections | 5746 ($R_{int}$ = 0.0267) |
| Absorption correction | USED SADABS |
| Max. and min. transmission | 0.978 and 0.772 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 5489 / 0 / 488 |
| Goodness-of-fit on $F^2$ | 1.041 |
| Final R indices [I>2σ(I)] | R1 = 0.0436, wR2 = 0.0947 |
| R indices (all data) | R1 = 0.0639, wR2 = 0.1117 |
| Absolute structure parameter | -2.2(12) |
| Extinction coefficient | 0.0046(5) |
| Largest diff. peak and hole | 0.177 and -0.179 eÅ$^{-3}$ |

Table 2. Bond lengths [Å] and angles [°] for IC6498.

| | | | |
|---|---|---|---|
| O(1)-C(1) | 1.217(3) | O(2)-C(17) | 1.223(3) |
| O(3)-C(20) | 1.440(3) | N(1)-C(1) | 1.377(4) |
| N(1)-C(11) | 1.423(3) | N(1)-N(2) | 1.433(3) |
| N(2)-C(17) | 1.374(3) | N(2)-C(3) | 1.476(3) |
| C(1)-C(2) | 1.488(4) | C(2)-C(7) | 1.523(4) |
| C(2)-C(3) | 1.538(4) | C(2)-C(8) | 1.565(4) |
| C(3)-C(4) | 1.549(4) | C(4)-C(5) | 1.529(4) |
| C(5)-C(8) | 1.542(4) | C(5)-C(6) | 1.542(4) |
| C(6)-C(7) | 1.552(4) | C(8)-C(10) | 1.527(4) |
| C(8)-C(9) | 1.548(4) | C(11)-C(12) | 1.375(4) |
| C(11)-C(16) | 1.381(4) | C(12)-C(13) | 1.379(5) |
| C(13)-C(14) | 1.380(5) | C(14)-C(15) | 1.358(5) |
| C(15)-C(16) | 1.371(4) | C(17)-C(18) | 1.503(4) |
| C(18)-C(19) | 1.302(4) | C(18)-C(20) | 1.517(4) |
| C(20)-C(21) | 1.511(4) | C(21)-C(22) | 1.500(4) |
| O(4)-C(23) | 1.216(3) | O(5)-C(39) | 1.234(3) |
| O(6)-C(42) | 1.427(3) | N(3)-C(23) | 1.390(4) |
| N(3)-N(4) | 1.420(3) | N(3)-C(33) | 1.435(3) |
| N(4)-C(39) | 1.366(3) | N(4)-C(25) | 1.486(3) |
| C(23)-C(24) | 1.487(4) | C(24)-C(25) | 1.533(4) |
| C(24)-C(29) | 1.537(4) | C(24)-C(30) | 1.571(4) |
| C(25)-C(26) | 1.542(4) | C(26)-C(27) | 1.540(4) |
| C(27)-C(30) | 1.541(4) | C(27)-C(28) | 1.541(4) |
| C(28)-C(29) | 1.561(4) | C(30)-C(32) | 1.528(4) |
| C(30)-C(31) | 1.542(4) | C(33)-C(38) | 1.367(4) |
| C(33)-C(34) | 1.382(4) | C(34)-C(35) | 1.387(5) |
| C(35)-C(36) | 1.354(5) | C(36)-C(37) | 1.372(5) |
| C(37)-C(38) | 1.386(4) | C(39)-C(40) | 1.491(4) |
| C(40)-C(41) | 1.317(4) | C(40)-C(42) | 1.512(4) |
| C(42)-C(43) | 1.511(4) | C(43)-C(44) | 1.525(5) |

| | | | |
|---|---|---|---|
| C(1)-N(1)-C(11) | 123.0(2) | C(1)-N(1)-N(2) | 111.8(2) |
| C(11)-N(1)-N(2) | 118.3(2) | C(17)-N(2)-N(1) | 116.1(2) |
| C(17)-N(2)-C(3) | 131.1(2) | N(1)-N(2)-C(3) | 107.4(2) |
| O(1)-C(1)-N(1) | 123.3(3) | O(1)-C(1)-C(2) | 129.4(3) |
| N(1)-C(1)-C(2) | 107.3(2) | C(1)-C(2)-C(7) | 121.2(2) |
| C(1)-C(2)-C(3) | 104.6(2) | C(7)-C(2)-C(3) | 106.6(2) |
| C(1)-C(2)-C(8) | 116.6(2) | C(7)-C(2)-C(8) | 102.0(2) |
| C(3)-C(2)-C(8) | 104.4(2) | N(2)-C(3)-C(2) | 103.2(2) |
| N(2)-C(3)-C(4) | 121.1(2) | C(2)-C(3)-C(4) | 103.0(2) |
| C(5)-C(4)-C(3) | 102.2(2) | C(4)-C(5)-C(8) | 102.0(2) |
| C(4)-C(5)-C(6) | 109.2(2) | C(8)-C(5)-C(6) | 101.9(2) |
| C(5)-C(6)-C(7) | 104.1(2) | C(2)-C(7)-C(6) | 101.4(2) |
| C(10)-C(8)-C(5) | 115.0(3) | C(10)-C(8)-C(9) | 106.5(2) |
| C(5)-C(8)-C(9) | 115.3(2) | C(10)-C(8)-C(2) | 115.8(2) |
| C(5)-C(8)-C(2) | 91.8(2) | C(9)-C(8)-C(2) | 112.3(2) |
| C(12)-C(11)-C(16) | 120.2(3) | C(12)-C(11)-N(1) | 120.9(3) |
| C(16)-C(11)-N(1) | 118.9(3) | C(11)-C(12)-C(13) | 119.7(3) |
| C(14)-C(13)-C(12) | 119.6(3) | C(15)-C(14)-C(13) | 120.4(3) |
| C(14)-C(15)-C(16) | 120.5(3) | C(15)-C(16)-C(11) | 119.5(3) |
| O(2)-C(17)-N(2) | 121.8(3) | O(2)-C(17)-C(18) | 122.3(3) |
| N(2)-C(17)-C(18) | 115.5(3) | C(19)-C(18)-C(17) | 120.1(3) |
| C(19)-C(18)-C(20) | 122.4(3) | C(17)-C(18)-C(20) | 117.3(2) |
| O(3)-C(20)-C(21) | 112.3(2) | O(3)-C(20)-C(18) | 106.5(2) |
| C(21)-C(20)-C(18) | 116.0(2) | C(22)-C(21)-C(20) | 113.1(3) |
| C(23)-N(3)-N(4) | 111.3(2) | C(23)-N(3)-C(33) | 122.5(2) |
| N(4)-N(3)-C(33) | 118.2(2) | C(39)-N(4)-N(3) | 118.0(2) |
| C(39)-N(4)-C(25) | 129.9(2) | N(3)-N(4)-C(25) | 107.8(2) |
| O(4)-C(23)-N(3) | 124.0(3) | O(4)-C(23)-C(24) | 128.4(3) |

| | | | |
|---|---|---|---|
| N(3)-C(23)-C(24) | 107.6(2) | C(23)-C(24)-C(25) | 104.8(2) |
| C(23)-C(24)-C(29) | 120.8(2) | C(25)-C(24)-C(29) | 105.7(2) |
| C(23)-C(24)-C(30) | 117.5(2) | C(25)-C(24)-C(30) | 104.6(2) |
| C(29)-C(24)-C(30) | 101.9(2) | N(4)-C(25)-C(24) | 102.8(2) |
| N(4)-C(25)-C(26) | 120.6(2) | C(24)-C(25)-C(26) | 103.7(2) |
| C(27)-C(26)-C(25) | 101.4(2) | C(26)-C(27)-C(30) | 101.8(2) |
| C(26)-C(27)-C(28) | 109.5(3) | C(30)-C(27)-C(28) | 102.9(2) |
| C(27)-C(28)-C(29) | 103.7(2) | C(24)-C(29)-C(28) | 101.2(2) |
| C(32)-C(30)-C(31) | 107.0(3) | C(32)-C(30)-C(27) | 115.5(2) |
| C(31)-C(30)-C(27) | 113.9(2) | C(32)-C(30)-C(24) | 115.1(2) |
| C(31)-C(30)-C(24) | 113.6(2) | C(27)-C(30)-C(24) | 91.5(2) |
| C(38)-C(33)-C(34) | 120.2(3) | C(38)-C(33)-N(3) | 121.4(3) |
| C(34)-C(33)-N(3) | 118.2(3) | C(33)-C(34)-C(35) | 119.2(3) |
| C(36)-C(35)-C(34) | 120.6(3) | C(35)-C(36)-C(37) | 120.1(3) |
| C(36)-C(37)-C(38) | 120.2(3) | C(33)-C(38)-C(37) | 119.7(3) |
| O(5)-C(39)-N(4) | 121.9(2) | O(5)-C(39)-C(40) | 121.6(3) |
| N(4)-C(39)-C(40) | 116.2(2) | C(41)-C(40)-C(39) | 119.9(3) |
| C(41)-C(40)-C(42) | 121.6(3) | C(39)-C(40)-C(42) | 118.3(2) |
| O(6)-C(42)-C(40) | 110.5(2) | O(6)-C(42)-C(43) | 111.9(2) |
| C(40)-C(42)-C(43) | 113.2(2) | C(42)-C(43)-C(44) | 112.5(3) |

Symmetry transformations used to generate equivalent atoms:

displacement parameters [&GA&^2&0 x 10&^3&0] for IC6498. U(eq) is defined as one third of the trace of the orthogonalized &IU&vij&0&:&! tensor.&N

| | x | y | z | U(eq) |
|---|---|---|---|---|
| O(1) | -4960(2) | 2919(2) | 315(1) | 67(1) |
| O(2) | -1375(2) | 106(2) | 788(1) | 61(1) |
| O(3) | 950(2) | 1721(2) | 1247(1) | 63(1) |
| N(1) | -2928(2) | 1756(2) | 440(1) | 47(1) |
| N(2) | -1466(2) | 2054(2) | 514(1) | 44(1) |
| C(1) | -3734(3) | 2915(3) | 409(1) | 46(1) |
| C(2) | -2827(3) | 4069(3) | 512(1) | 46(1) |
| C(3) | -1283(3) | 3534(3) | 491(1) | 46(1) |
| C(4) | -497(3) | 4342(3) | 744(1) | 57(1) |
| C(5) | -1624(3) | 5387(3) | 836(1) | 56(1) |
| C(6) | -1944(4) | 6317(3) | 562(1) | 68(1) |
| C(7) | -2849(4) | 5420(3) | 344(1) | 61(1) |
| C(8) | -2995(3) | 4521(3) | 852(1) | 52(1) |
| C(9) | -4398(4) | 5318(3) | 908(1) | 72(1) |
| C(10) | -2962(4) | 3385(3) | 1089(1) | 68(1) |
| C(11) | -3231(3) | 541(3) | 278(1) | 46(1) |
| C(12) | -2413(3) | 157(3) | 31(1) | 59(1) |
| C(13) | -2766(4) | -997(4) | -130(1) | 71(1) |
| C(14) | -3940(5) | -1750(3) | -42(1) | 77(1) |
| C(15) | -4750(4) | -1359(3) | 200(1) | 73(1) |
| C(16) | -4403(3) | -222(3) | 364(1) | 58(1) |
| C(17) | -763(3) | 1105(3) | 688(1) | 45(1) |
| C(18) | 829(3) | 1295(3) | 714(1) | 45(1) |
| C(19) | 1568(3) | 1719(3) | 481(1) | 65(1) |
| C(20) | 1530(3) | 862(3) | 1012(1) | 51(1) |
| C(21) | 1408(4) | -622(3) | 1091(1) | 63(1) |
| C(22) | 2031(5) | -1533(4) | 852(1) | 94(1) |
| O(4) | 4920(2) | -2769(2) | 2075(1) | 65(1) |
| O(5) | 2148(2) | 1271(2) | 1840(1) | 52(1) |
| O(6) | 3518(2) | 3213(2) | 1381(1) | 67(1) |
| N(3) | 3867(2) | -661(2) | 2072(1) | 44(1) |
| N(4) | 4319(2) | 702(2) | 2038(1) | 40(1) |
| C(23) | 5002(3) | -1555(3) | 2033(1) | 46(1) |
| C(24) | 6272(3) | -748(3) | 1938(1) | 43(1) |
| C(25) | 5910(3) | 710(3) | 2030(1) | 40(1) |
| C(26) | 6718(3) | 1574(3) | 1792(1) | 52(1) |
| C(27) | 7679(3) | 503(3) | 1639(1) | 56(1) |
| C(28) | 8722(3) | -86(3) | 1878(1) | 66(1) |
| C(29) | 7757(3) | -1034(3) | 2075(1) | 57(1) |
| C(30) | 6616(3) | -668(3) | 1587(1) | 52(1) |
| C(31) | 7311(4) | -1964(4) | 1459(1) | 77(1) |
| C(32) | 5336(3) | -339(3) | 1383(1) | 67(1) |
| C(33) | 2694(3) | -937(3) | 2275(1) | 44(1) |
| C(34) | 1892(3) | -2092(3) | 2225(1) | 60(1) |
| C(35) | 834(4) | -2441(4) | 2434(1) | 77(1) |
| C(36) | 568(4) | -1648(4) | 2680(1) | 77(1) |
| C(37) | 1349(4) | -489(4) | 2726(1) | 70(1) |
| C(38) | 2423(3) | -132(3) | 2522(1) | 53(1) |
| C(39) | 3363(3) | 1611(3) | 1921(1) | 41(1) |
| C(40) | 3808(3) | 3060(3) | 1929(1) | 42(1) |
| C(41) | 4428(4) | 3557(3) | 2175(1) | 66(1) |
| C(42) | 3414(3) | 3951(3) | 1661(1) | 51(1) |
| C(43) | 1970(4) | 4623(3) | 1696(1) | 72(1) |

Table 2. Anisotropic displacement parameters [&GA&^2&0 x 10&^3&0] for IC6498.&NThe anisotropic displacement factor exponent takes the form:&N -2&Gp&^2&0 [ (ha&^*&0)&^2&0U&v11&0 + ... + 2hka&^*&0b&^*&0U&v12&0

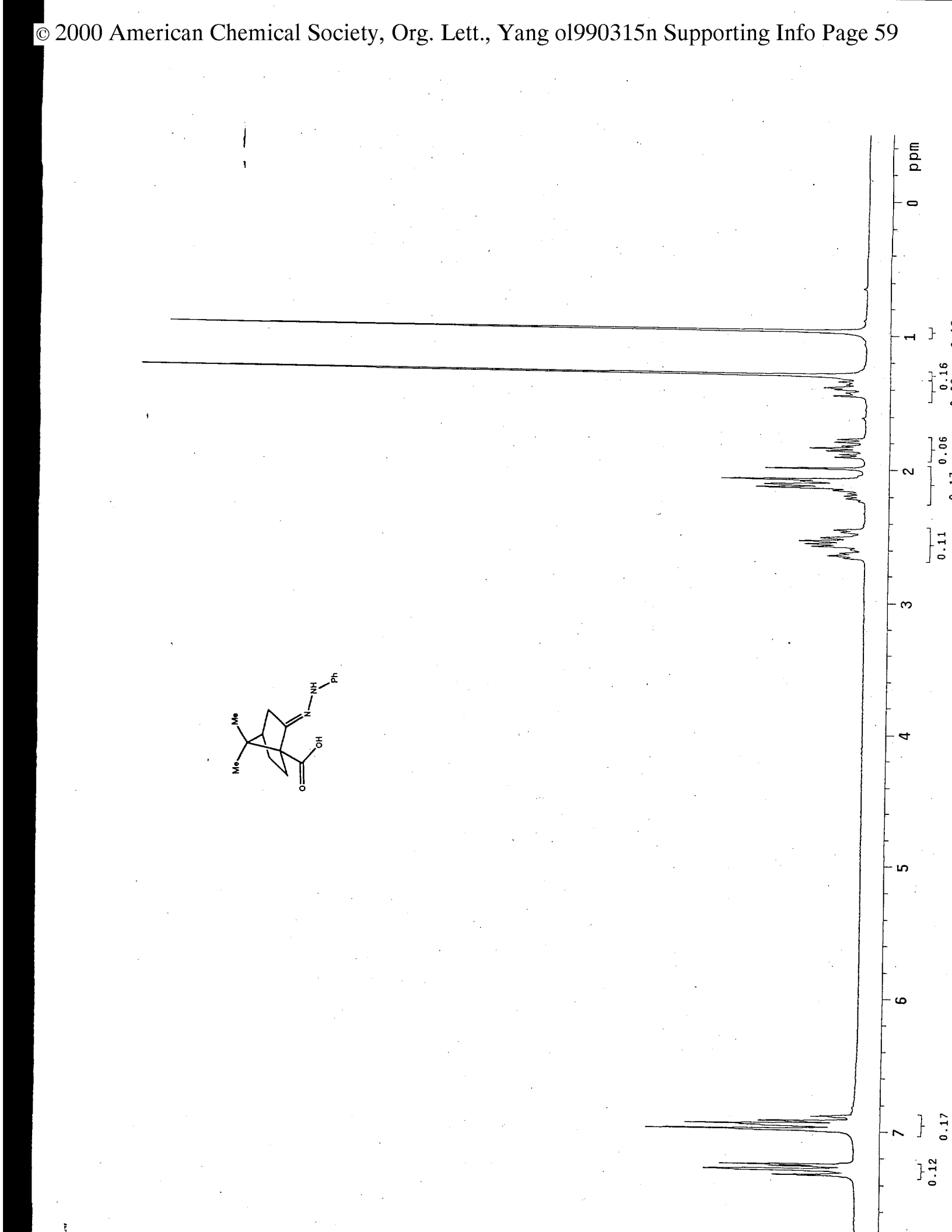
| | | | | | | |
|---|---|---|---|---|---|---|
| O(1) | 43(1) | 63(1) | 95(2) | 9(1) | -16(1) | 7(1) |
| O(2) | 45(1) | 63(1) | 75(1) | 21(1) | -12(1) | -8(1) |
| O(3) | 63(1) | 74(1) | 52(1) | -6(1) | -2(1) | 7(1) |
| N(1) | 35(1) | 44(1) | 61(2) | 4(1) | -9(1) | 2(1) |
| N(2) | 32(1) | 45(1) | 57(1) | 4(1) | -6(1) | -3(1) |
| C(1) | 38(2) | 48(2) | 52(2) | 9(1) | 1(2) | 0(2) |
| C(2) | 45(2) | 43(2) | 51(2) | 10(1) | 2(1) | 1(1) |
| C(3) | 43(2) | 46(2) | 48(2) | 7(1) | 5(1) | -4(1) |
| C(4) | 49(2) | 55(2) | 66(2) | -2(2) | 2(2) | -9(2) |
| C(5) | 59(2) | 51(2) | 59(2) | -4(2) | 3(2) | -2(2) |
| C(6) | 69(2) | 51(2) | 84(2) | 7(2) | 17(2) | -6(2) |
| C(7) | 67(2) | 52(2) | 64(2) | 14(2) | 4(2) | 3(2) |
| C(8) | 54(2) | 49(2) | 53(2) | 5(1) | 8(2) | 1(2) |
| C(9) | 70(2) | 62(2) | 86(2) | 0(2) | 25(2) | 11(2) |
| C(10) | 74(2) | 75(2) | 55(2) | 10(2) | 14(2) | 2(2) |
| C(11) | 39(2) | 41(2) | 59(2) | 5(2) | -9(1) | -3(1) |
| C(12) | 54(2) | 58(2) | 67(2) | 2(2) | -2(2) | 2(2) |
| C(13) | 83(3) | 64(2) | 66(2) | -7(2) | -8(2) | 25(2) |
| C(14) | 94(3) | 50(2) | 86(3) | -3(2) | -40(2) | 5(2) |
| C(15) | 61(2) | 62(2) | 95(3) | 5(2) | -16(2) | -21(2) |
| C(16) | 48(2) | 57(2) | 69(2) | 1(2) | -3(2) | -8(2) |
| C(17) | 42(2) | 53(2) | 39(2) | 3(2) | -4(1) | 0(2) |
| C(18) | 34(2) | 51(2) | 49(2) | -4(1) | -2(1) | -1(1) |
| C(19) | 40(2) | 88(2) | 67(2) | 10(2) | 0(2) | 4(2) |
| C(20) | 37(2) | 62(2) | 55(2) | -6(2) | -7(1) | 3(2) |
| C(21) | 59(2) | 68(2) | 61(2) | 7(2) | -13(2) | 10(2) |
| C(22) | 135(4) | 59(2) | 90(3) | -4(2) | -2(3) | 19(2) |
| O(4) | 65(1) | 35(1) | 94(2) | 5(1) | 5(1) | 1(1) |
| O(5) | 47(1) | 51(1) | 59(1) | 8(1) | -10(1) | -7(1) |
| O(6) | 82(2) | 69(1) | 50(1) | 9(1) | 10(1) | 0(1) |
| N(3) | 42(1) | 33(1) | 59(1) | 2(1) | 5(1) | -2(1) |
| N(4) | 40(1) | 30(1) | 51(1) | 4(1) | -2(1) | -3(1) |
| C(23) | 48(2) | 40(2) | 50(2) | 0(1) | -2(1) | 0(2) |
| C(24) | 40(2) | 39(2) | 49(2) | 3(1) | -2(1) | 1(1) |
| C(25) | 34(2) | 44(2) | 42(2) | 1(1) | -5(1) | -1(1) |
| C(26) | 43(2) | 52(2) | 61(2) | 4(2) | 4(2) | -6(2) |
| C(27) | 43(2) | 62(2) | 63(2) | 10(2) | 11(2) | -1(2) |
| C(28) | 42(2) | 71(2) | 85(2) | 2(2) | 3(2) | -1(2) |
| C(29) | 45(2) | 61(2) | 65(2) | 6(2) | 0(2) | 7(2) |
| C(30) | 51(2) | 54(2) | 50(2) | -3(2) | 4(1) | 3(2) |
| C(31) | 79(3) | 72(2) | 79(2) | -19(2) | 13(2) | 7(2) |
| C(32) | 68(2) | 84(2) | 49(2) | -10(2) | -7(2) | 1(2) |
| C(33) | 35(2) | 43(2) | 53(2) | 10(2) | -2(1) | 2(1) |
| C(34) | 55(2) | 54(2) | 71(2) | 5(2) | -7(2) | -13(2) |
| C(35) | 50(2) | 75(3) | 106(3) | 22(2) | -4(2) | -20(2) |
| C(36) | 54(2) | 81(3) | 97(3) | 34(2) | 16(2) | 6(2) |
| C(37) | 72(2) | 63(2) | 73(2) | 13(2) | 22(2) | 17(2) |
| C(38) | 45(2) | 47(2) | 67(2) | 8(2) | 8(2) | 4(2) |
| C(39) | 44(2) | 42(2) | 38(2) | 1(1) | -1(1) | -1(2) |
| C(40) | 41(2) | 40(2) | 47(2) | -1(1) | -1(1) | 2(1) |
| C(41) | 86(3) | 46(2) | 66(2) | -2(2) | -12(2) | 0(2) |

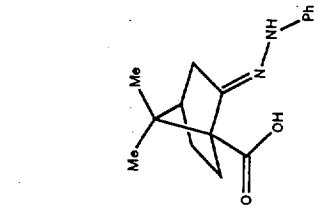
| | | | | | | |
|---|---|---|---|---|---|---|
| C(43) | 90(3) | 53(2) | 73(2) | -3(2) | -8(2) | 21(2) |
| C(44) | 158(4) | 82(3) | 98(3) | 13(2) | -41(3) | 41(3) |

Table 3. Hydrogen coordinates ( x 10&^4&0) and isotropic displacement parameters (&GA&^2&0 x 10&^3&0) for IC6498.&H

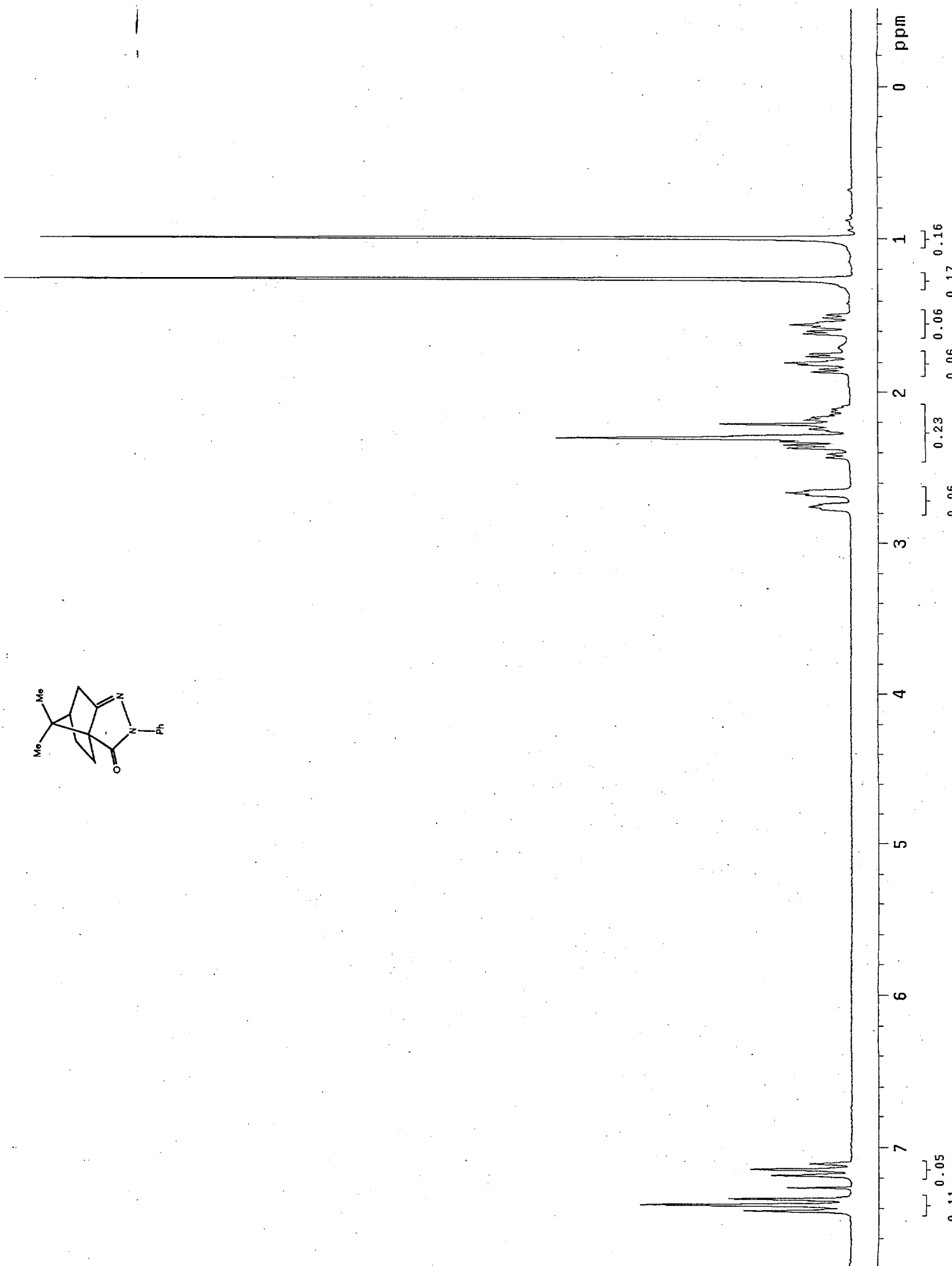
| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(3A) | 645(2) | 1253(2) | 1387(1) | 94 |
| H(3B) | -877(3) | 3765(3) | 291(1) | 55 |
| H(4A) | -240(3) | 3764(3) | 915(1) | 68 |
| H(4B) | 360(3) | 4774(3) | 665(1) | 68 |
| H(5A) | -1402(3) | 5873(3) | 1026(1) | 68 |
| H(6A) | -1064(4) | 6606(3) | 464(1) | 81 |
| H(6B) | -2479(4) | 7110(3) | 625(1) | 81 |
| H(7A) | -3816(4) | 5764(3) | 322(1) | 73 |
| H(7B) | -2410(4) | 5349(3) | 144(1) | 73 |
| H(9A) | -4466(4) | 6045(3) | 764(1) | 109 |
| H(9B) | -4396(4) | 5679(3) | 1112(1) | 109 |
| H(9C) | -5203(4) | 4722(3) | 884(1) | 109 |
| H(10A) | -3852(4) | 2899(3) | 1084(1) | 102 |
| H(10B) | -2826(4) | 3765(3) | 1289(1) | 102 |
| H(10C) | -2189(4) | 2778(3) | 1044(1) | 102 |
| H(12A) | -1625(3) | 673(3) | -27(1) | 71 |
| H(13A) | -2215(4) | -1265(4) | -296(1) | 85 |
| H(14A) | -4178(5) | -2531(3) | -149(1) | 92 |
| H(15A) | -5547(4) | -1867(3) | 256(1) | 87 |
| H(16A) | -4953(3) | 34(3) | 531(1) | 70 |
| H(19A) | 1110(3) | 1906(3) | 297(1) | 78 |
| H(19B) | 2553(3) | 1836(3) | 498(1) | 78 |
| H(20A) | 2553(3) | 1072(3) | 995(1) | 62 |
| H(21A) | 1896(4) | -784(3) | 1284(1) | 75 |
| H(21B) | 406(4) | -848(3) | 1119(1) | 75 |
| H(22A) | 1924(5) | -2459(4) | 914(1) | 142 |
| H(22B) | 3029(5) | -1330(4) | 826(1) | 142 |
| H(22C) | 1538(5) | -1393(4) | 662(1) | 142 |
| H(6C) | 3302(2) | 3708(2) | 1238(1) | 101 |
| H(25A) | 6285(3) | 897(3) | 2234(1) | 48 |
| H(26A) | 6066(3) | 1983(3) | 1646(1) | 62 |
| H(26B) | 7282(3) | 2277(3) | 1889(1) | 62 |
| H(27A) | 8148(3) | 819(3) | 1452(1) | 67 |
| H(28A) | 9140(3) | 626(3) | 2002(1) | 79 |
| H(28B) | 9484(3) | -592(3) | 1780(1) | 79 |
| H(29A) | 8026(3) | -1976(3) | 2051(1) | 69 |
| H(29B) | 7797(3) | -793(3) | 2290(1) | 69 |
| H(31A) | 8126(4) | -2201(4) | 1582(1) | 115 |
| H(31B) | 7614(4) | -1808(4) | 1252(1) | 115 |
| H(31C) | 6628(4) | -2689(4) | 1463(1) | 115 |
| H(32A) | 4873(3) | 467(3) | 1456(1) | 100 |
| H(32B) | 4669(3) | -1079(3) | 1387(1) | 100 |
| H(32C) | 5660(3) | -198(3) | 1177(1) | 100 |
| H(34A) | 2060(3) | -2630(3) | 2055(1) | 72 |
| H(35A) | 303(4) | -3226(4) | 2404(1) | 93 |
| H(36A) | -146(4) | -1888(4) | 2817(1) | 93 |
| H(37A) | 1158(4) | 57(4) | 2894(1) | 83 |
| H(38A) | 2956(3) | 650(3) | 2554(1) | 64 |

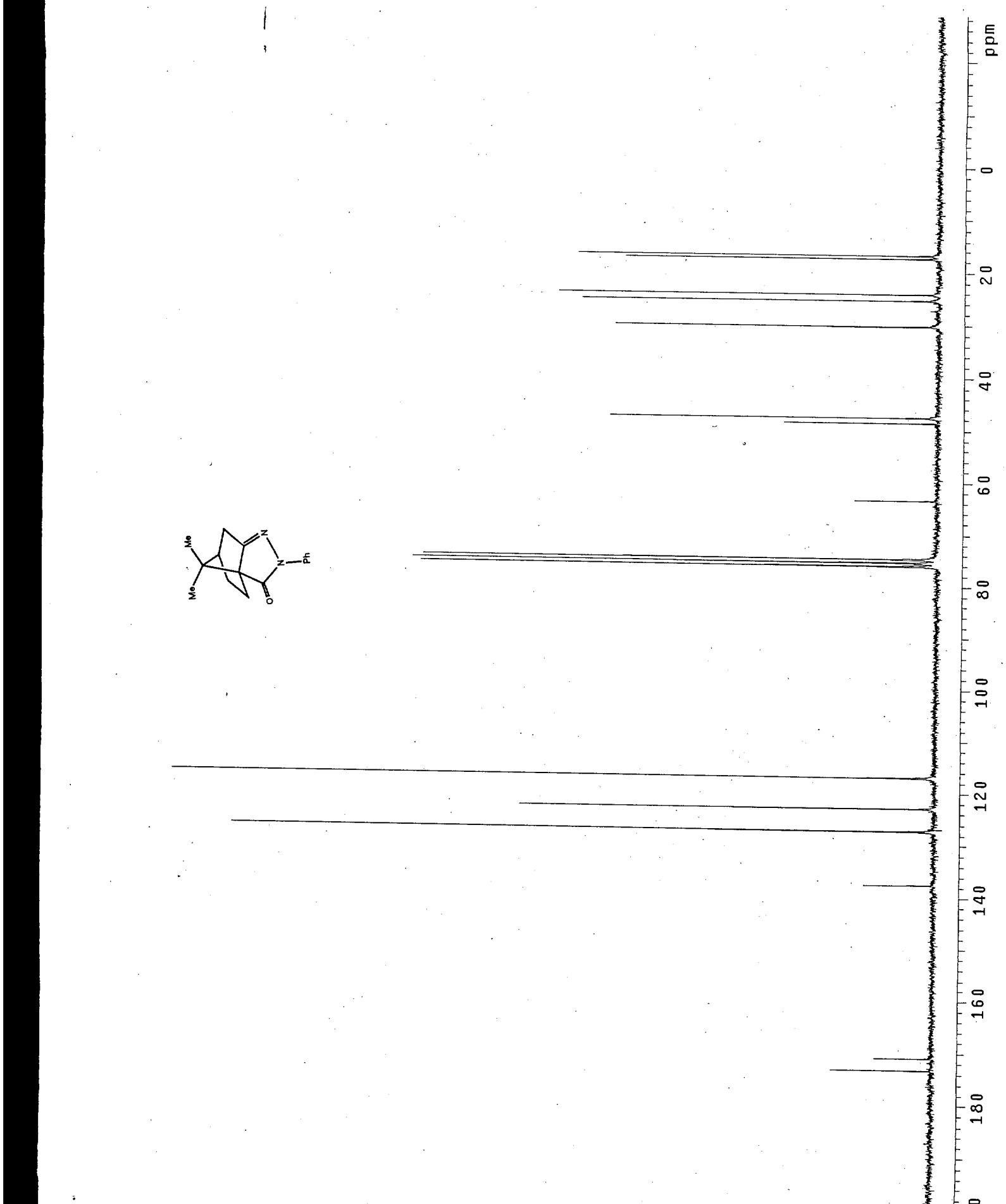
| | | | | |
|---|---|---|---|---|
| H(41B) | 4706(4) | 4460(3) | 2180(1) | 79 |
| H(42A) | 4130(3) | 4676(3) | 1652(1) | 61 |
| H(43A) | 1913(4) | 5038(3) | 1897(1) | 86 |
| H(43B) | 1227(4) | 3939(3) | 1683(1) | 86 |
| H(44A) | 772(5) | 6096(4) | 1485(1) | 169 |
| H(44B) | 1733(5) | 5291(4) | 1254(1) | 169 |
| H(44C) | 2421(5) | 6388(4) | 1467(1) | 169 |

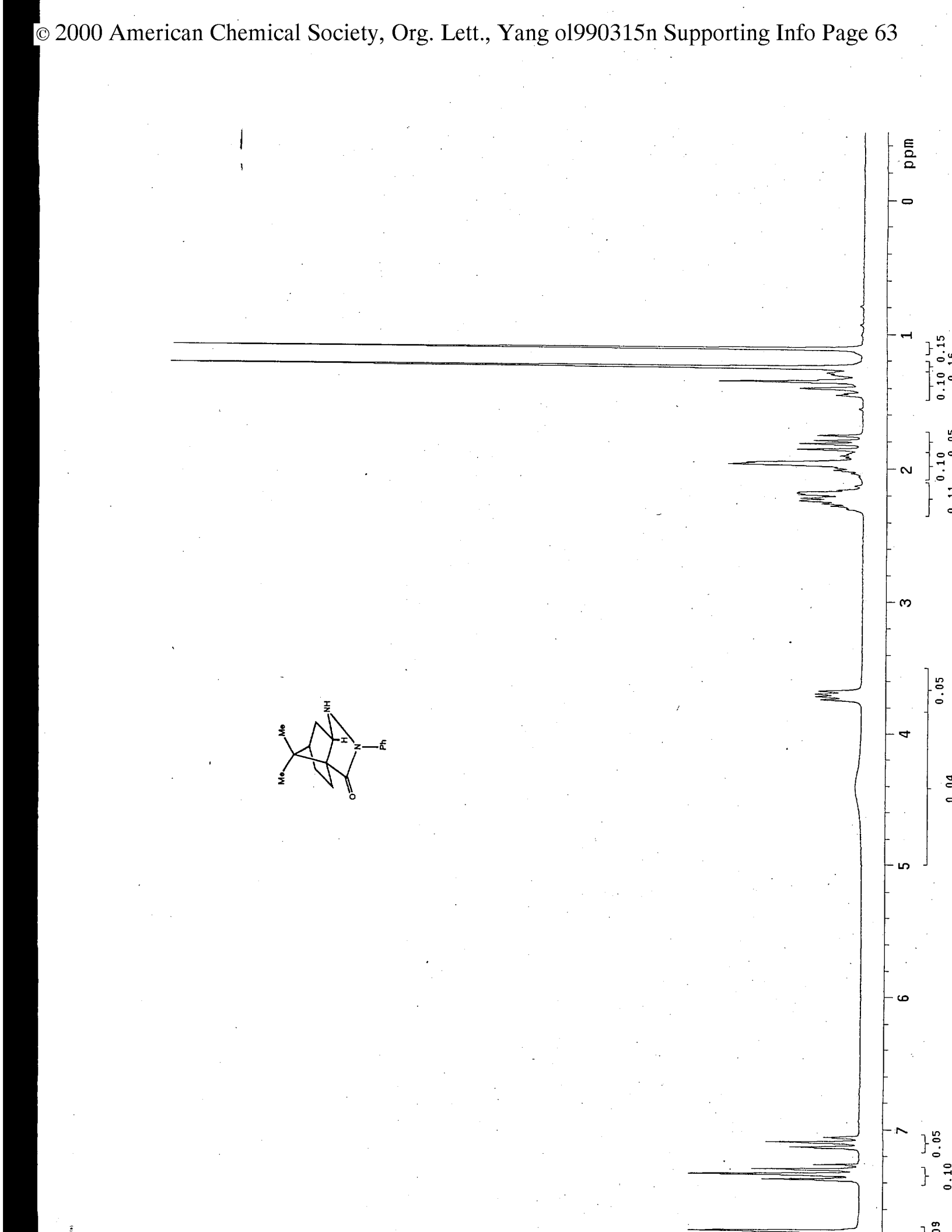


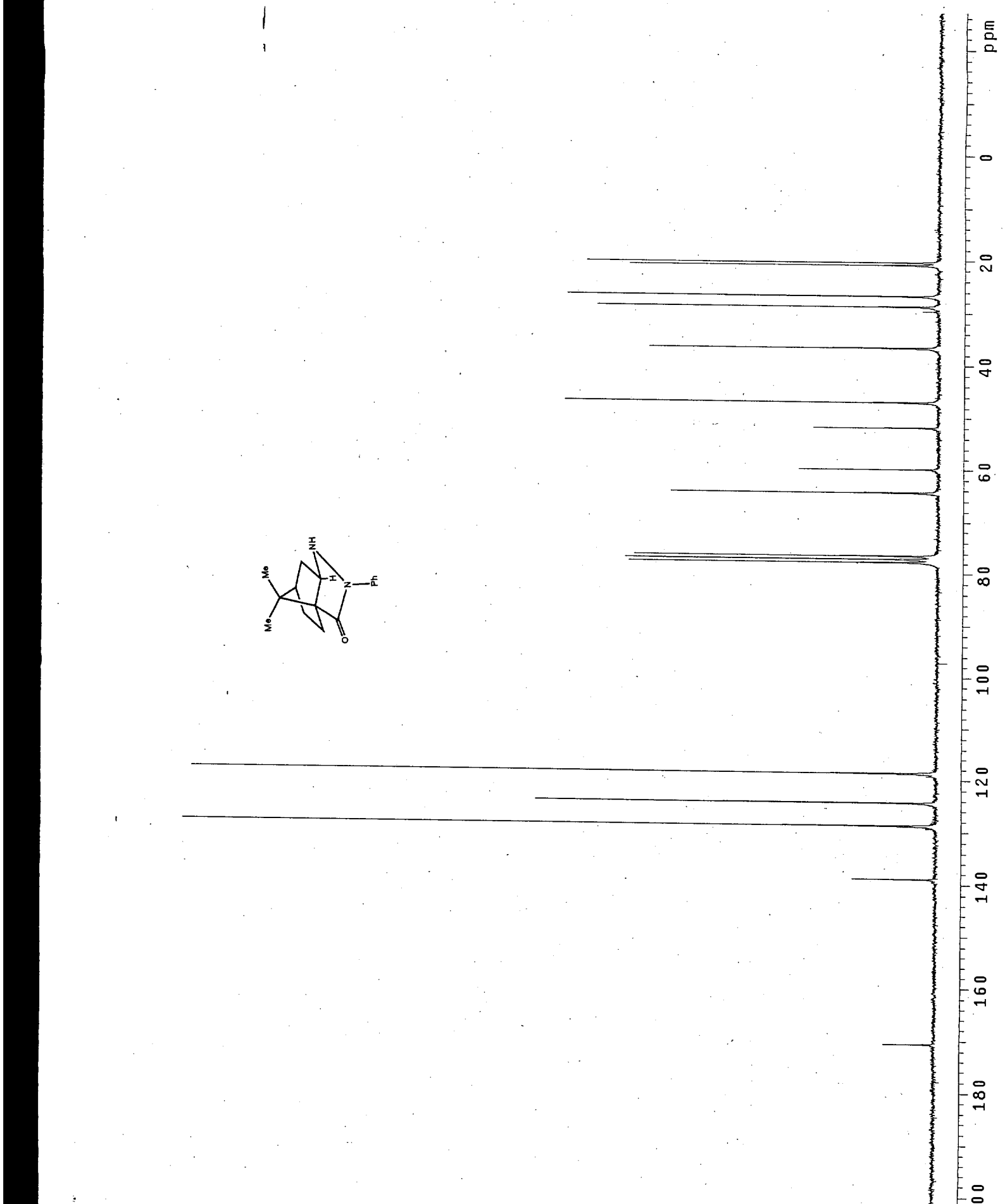

Me
Me
N
NH
Ph
OH
O

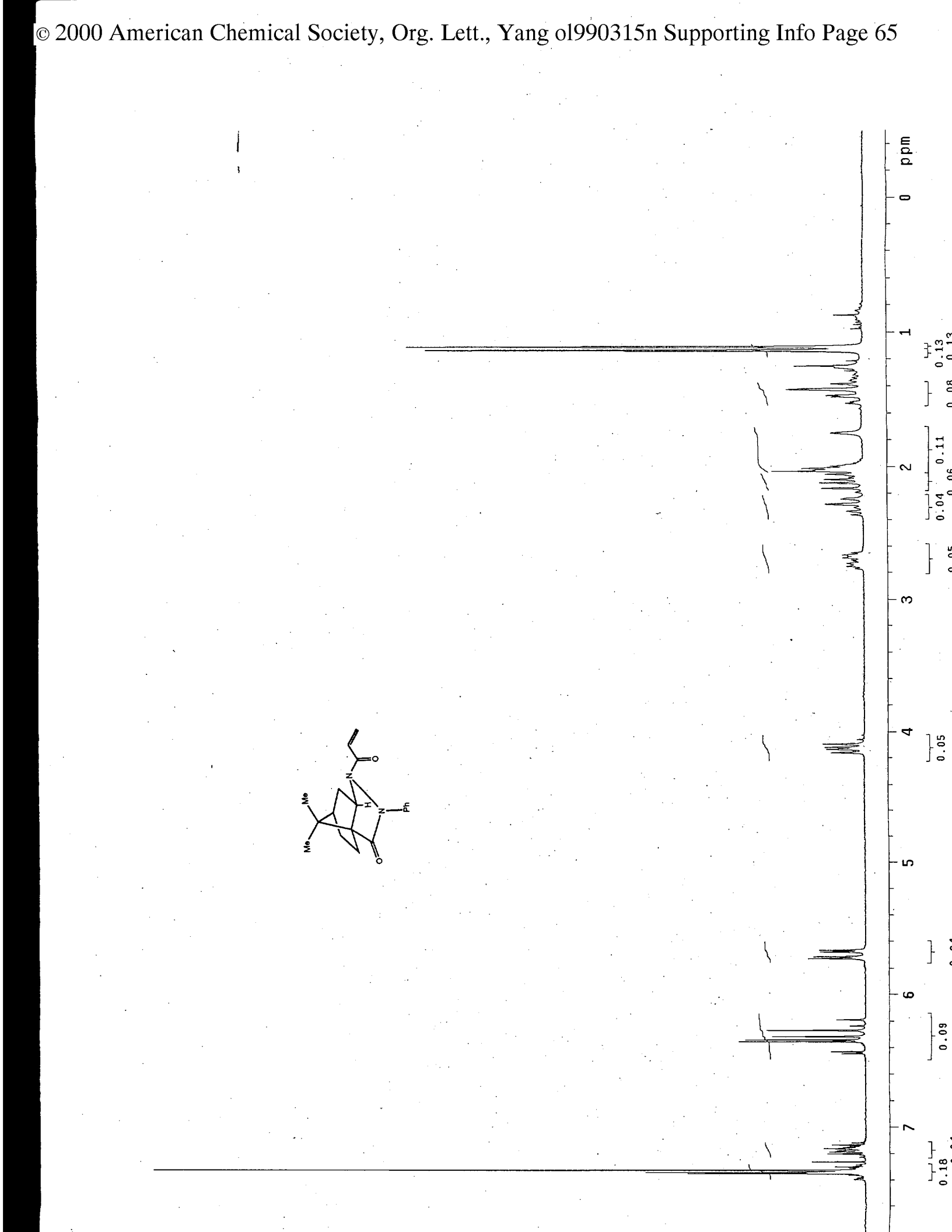

ppm
0
20
40
60
80
100
120
140
160
180
200

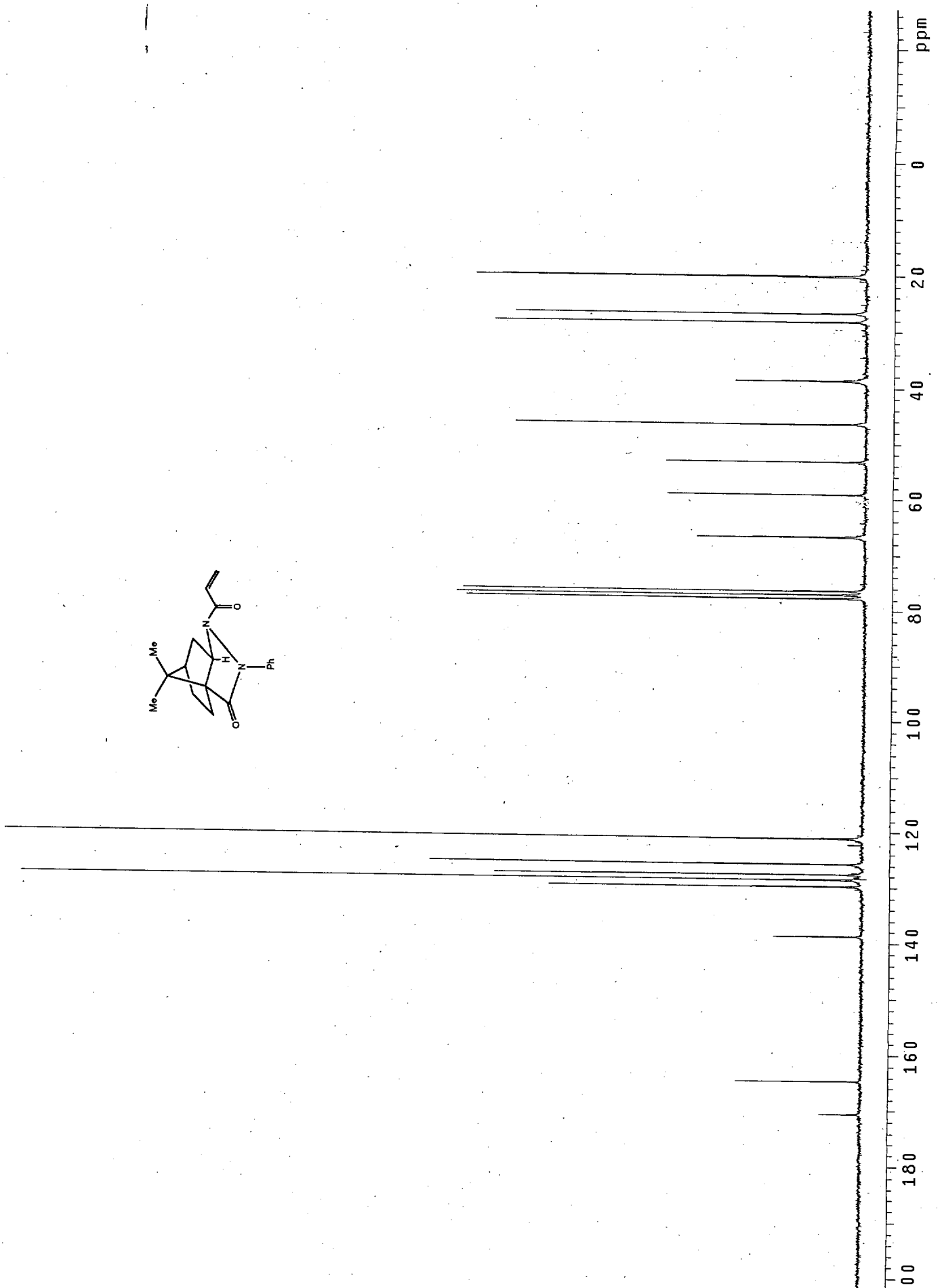

ppm
0
1
2
3
4
5
6
7
0.16
0.06
0.23
0.05
Me
Me
N
N
Ph
O

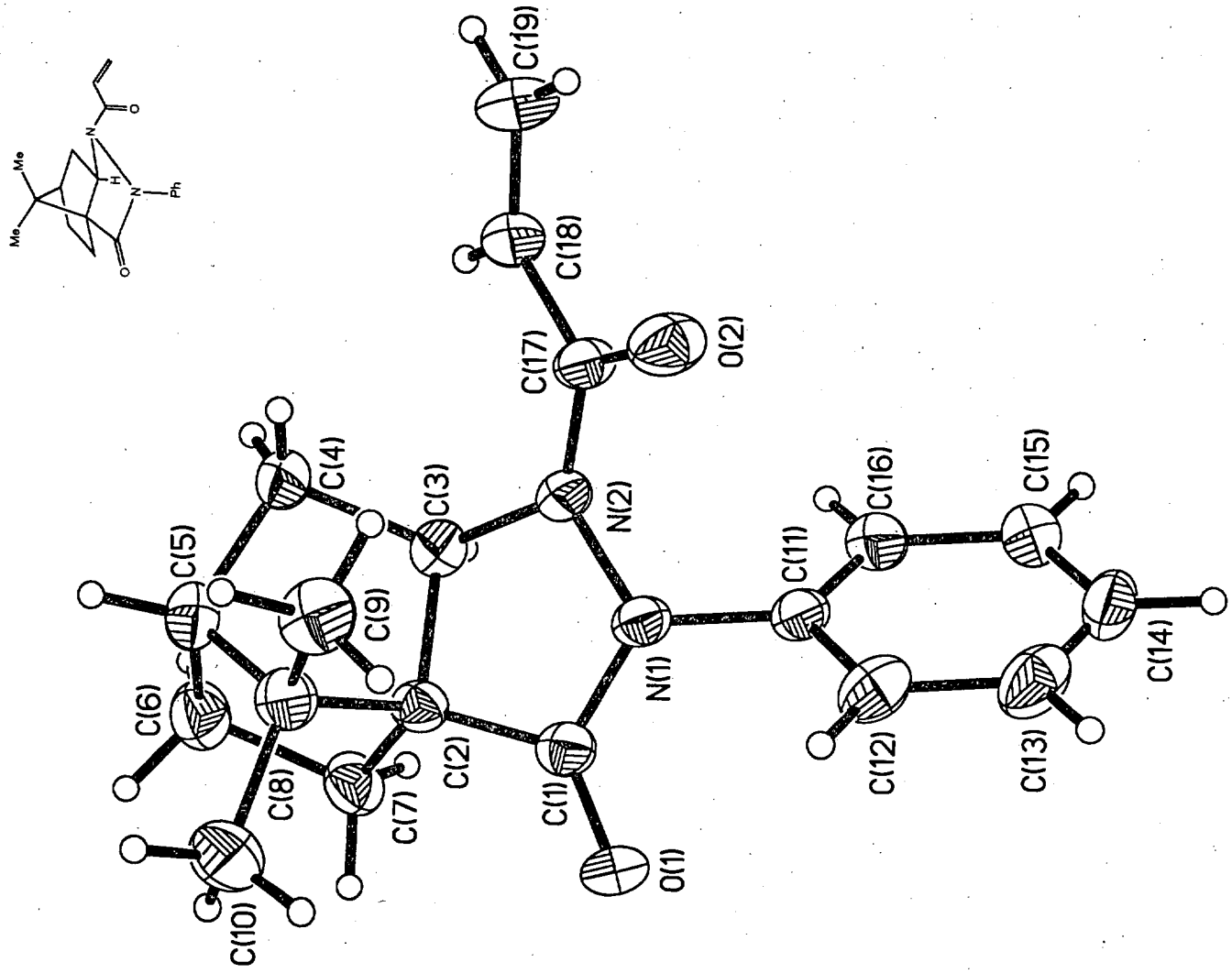


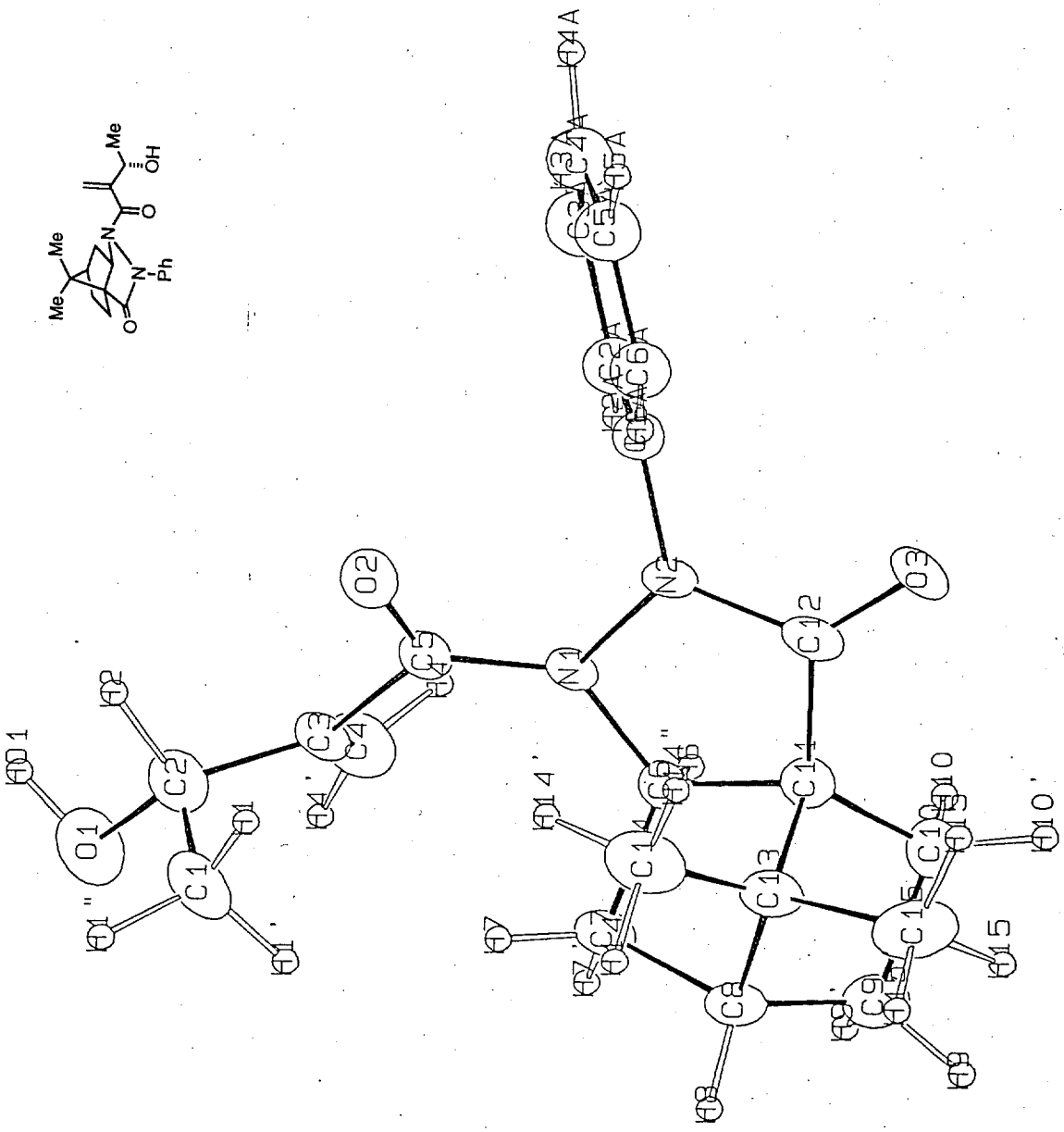


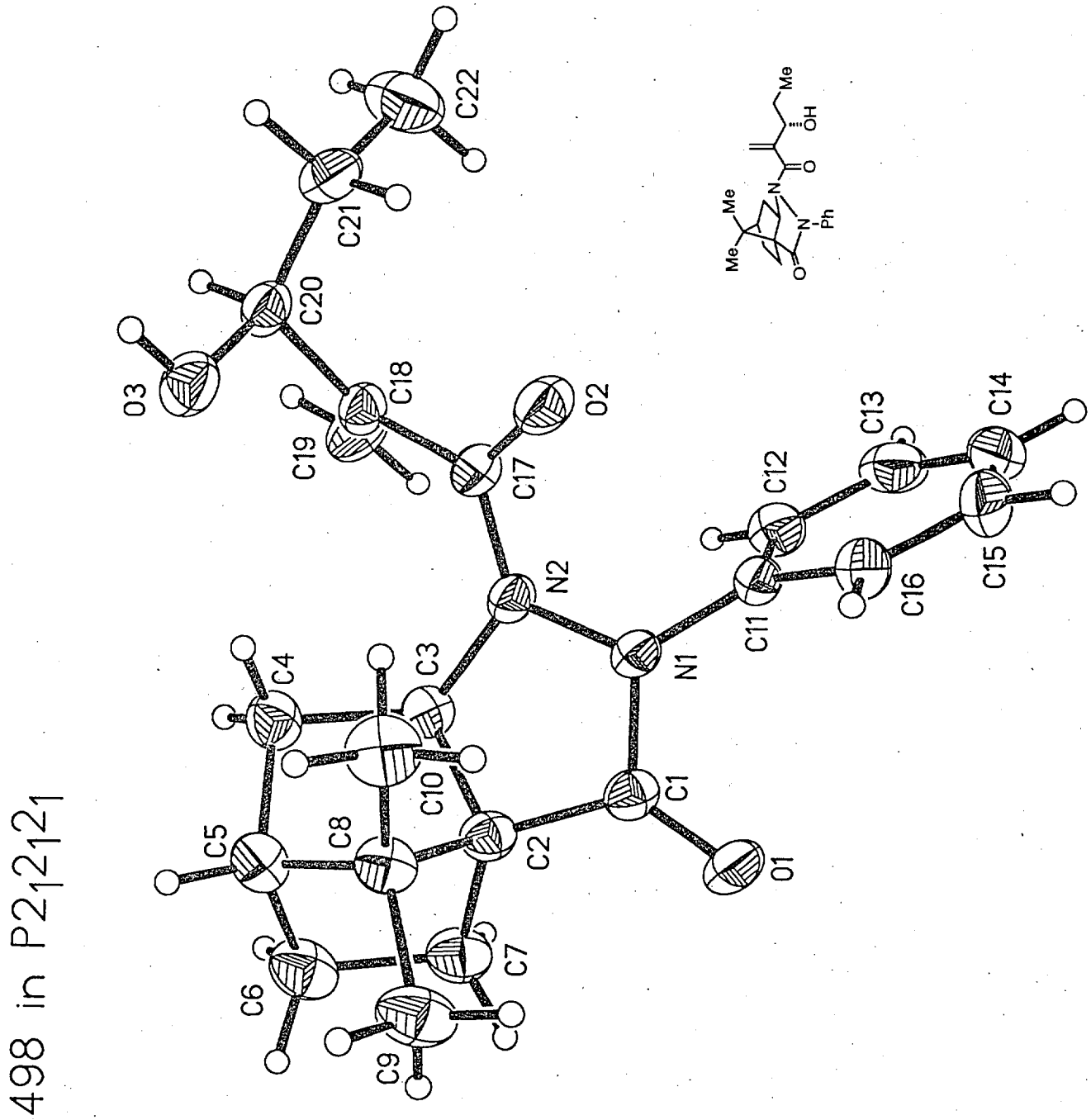







C(19)
C(18)
C(17)
O(2)
C(4)
C(3)
N(2)
C(16)
C(15)
C(5)
C(9)
C(11)
C(14)
C(6)
N(1)
C(8)
C(2)
C(7)
C(1)
C(12)
C(13)
O(1)
C(10)






IC6498 in P212121