Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

An Efficient Synthesis of 16*H*-Dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-ones *via* an Ullmann Reaction Catalyzed by CuI

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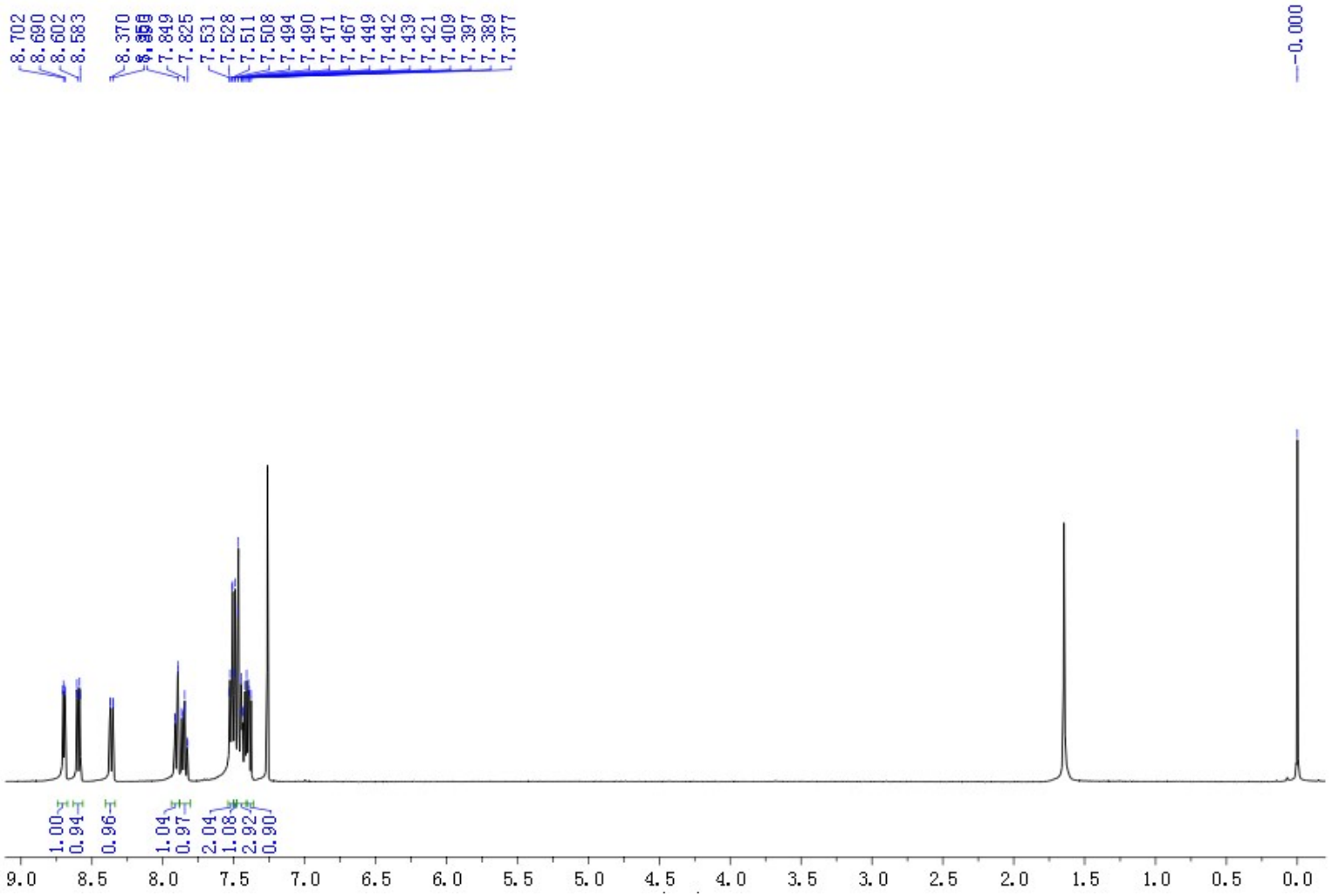
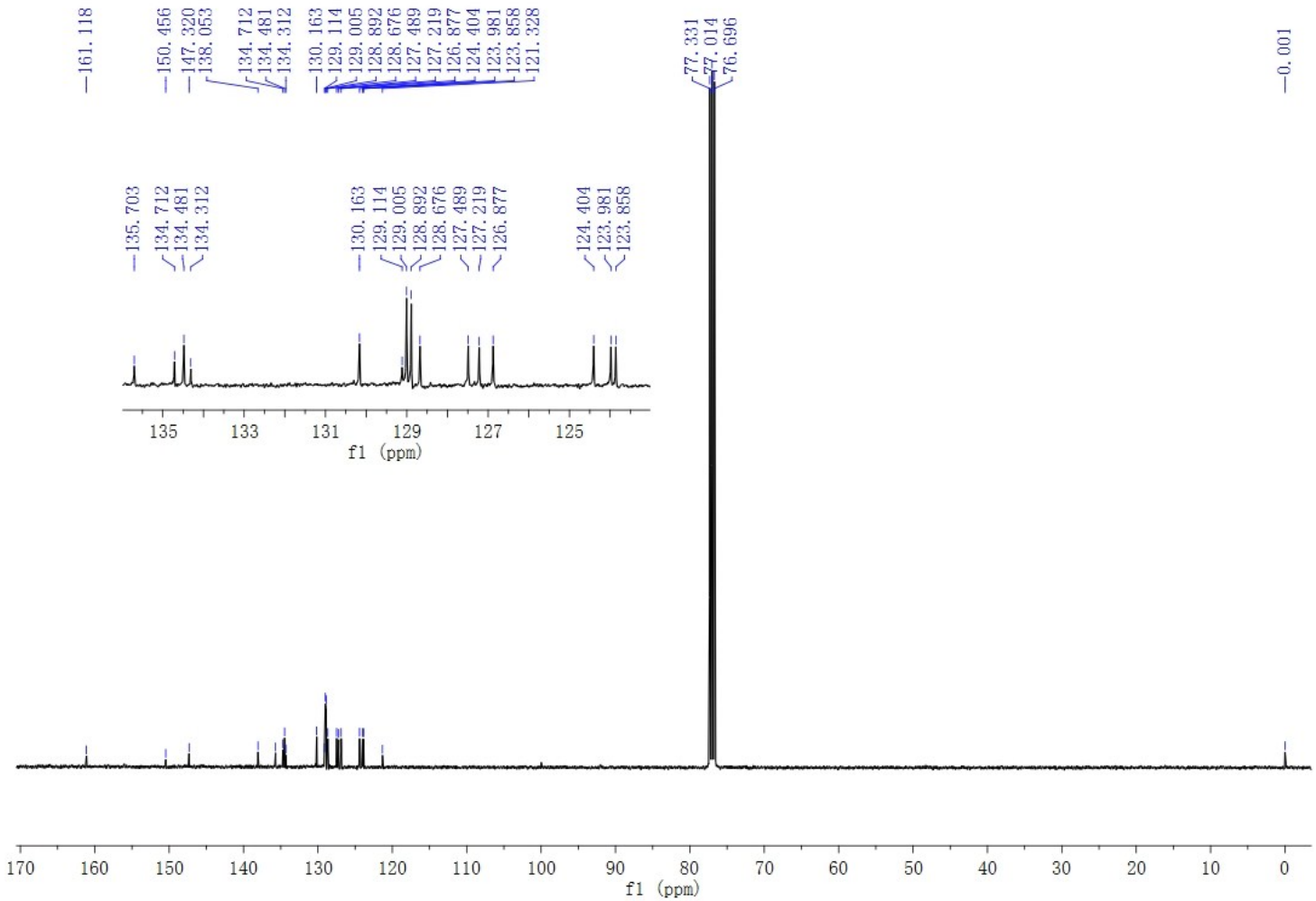
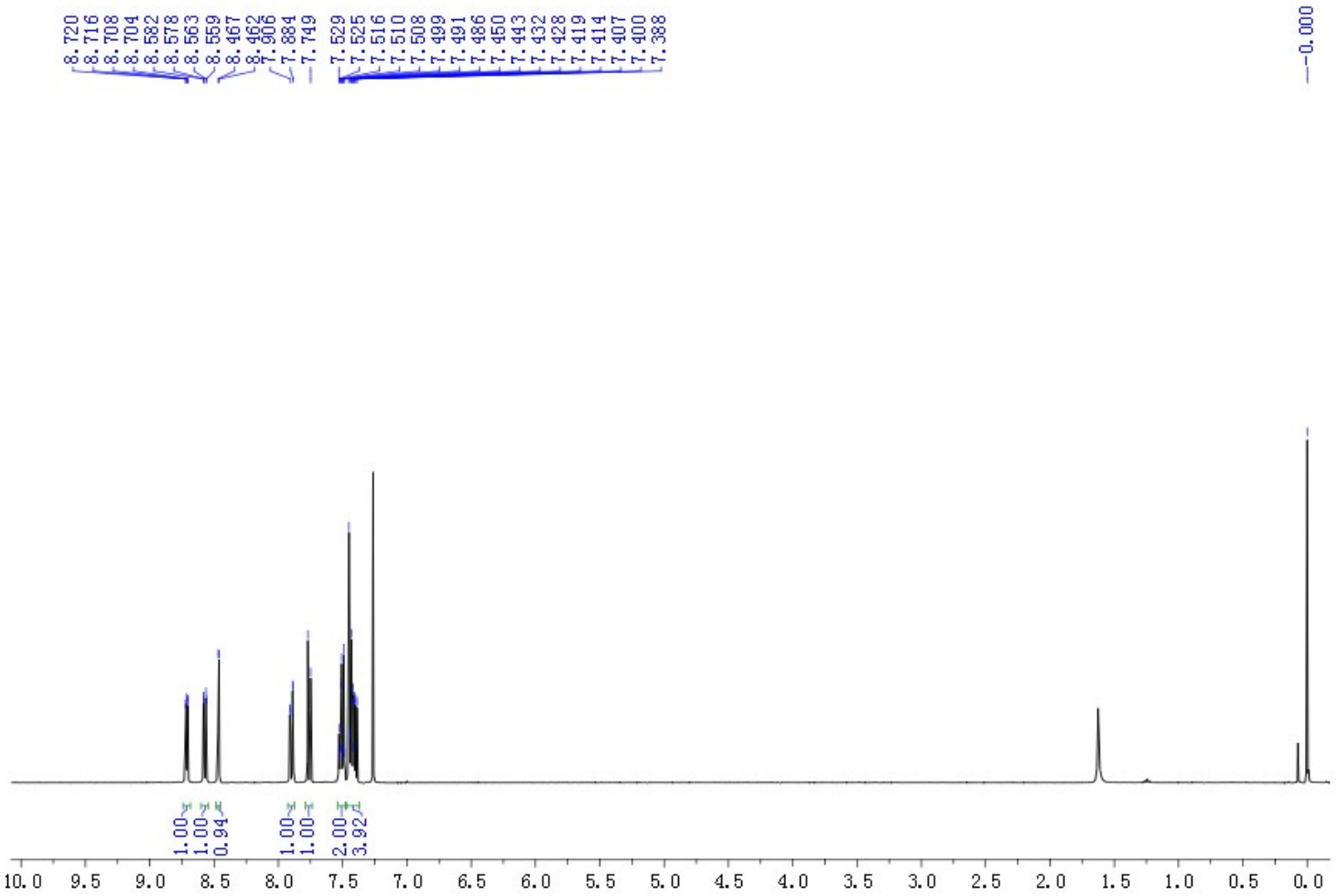
^{1}H and ^{13}C NMR spectra for compounds **3a-3u** S2-S43
CIF of the product **3c** S44-S61



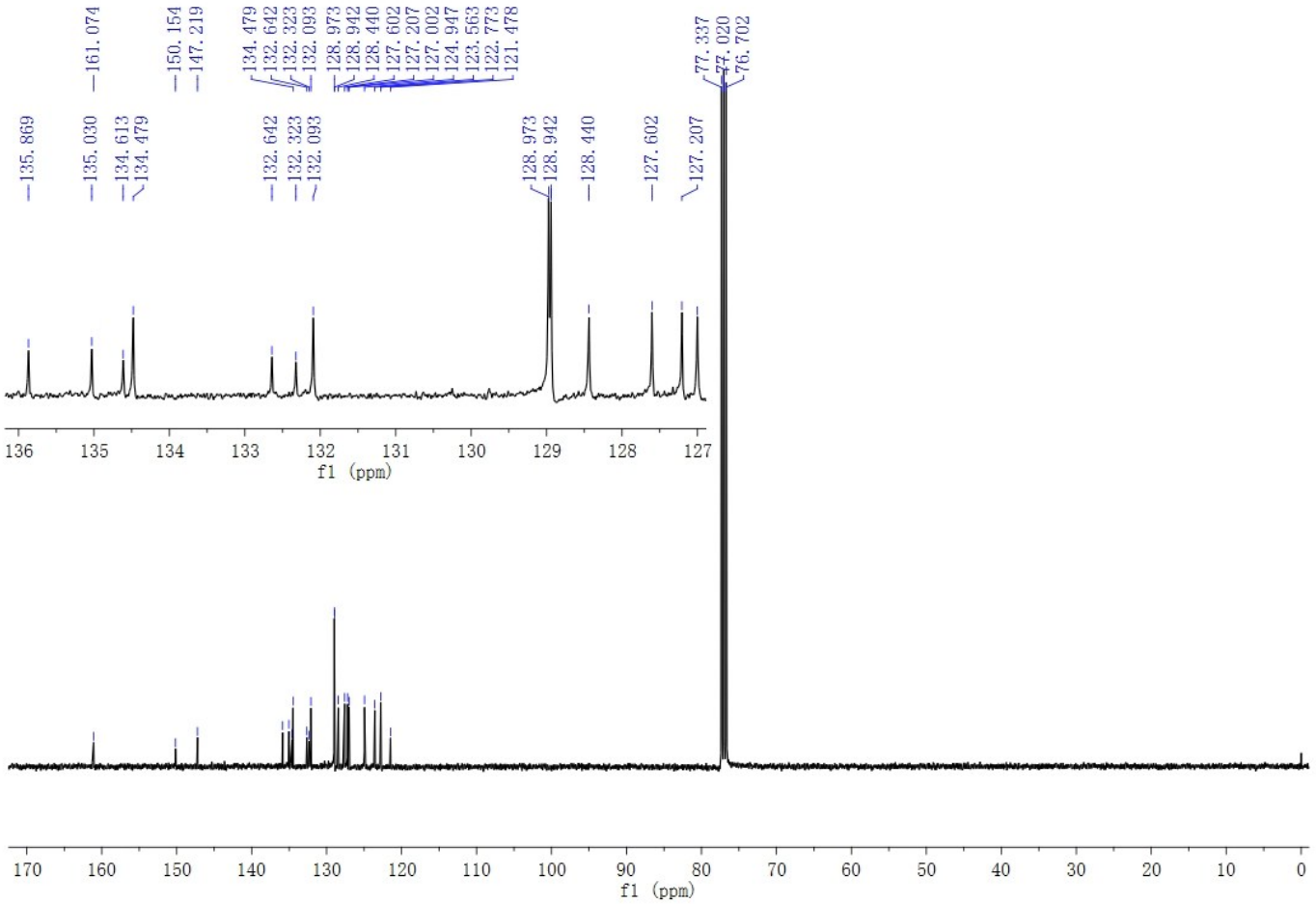
^{1}H NMR of 16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3a**)



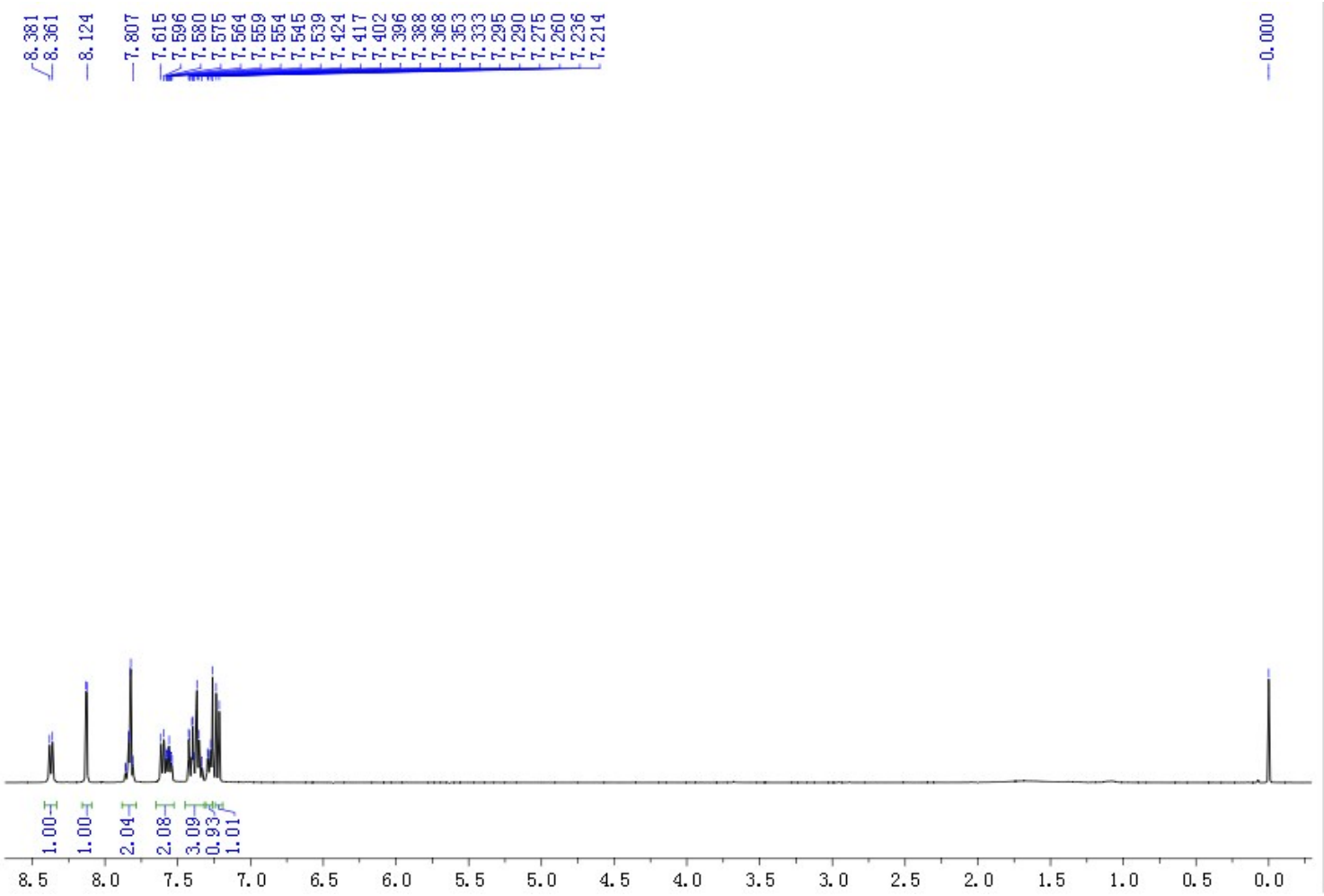
^{13}C NMR of 16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3a**)



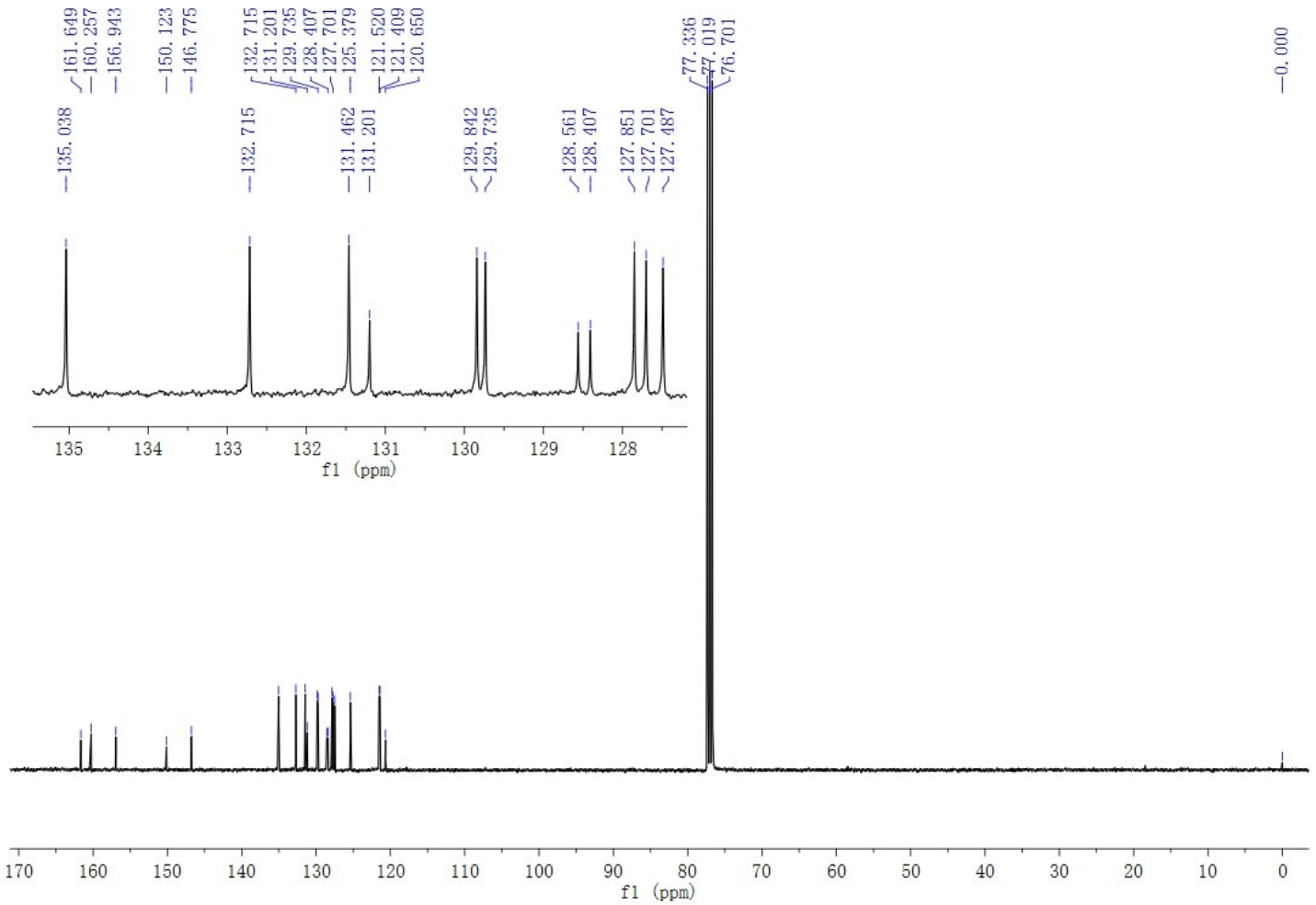
^{1}H NMR of 8-chloro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3b**)



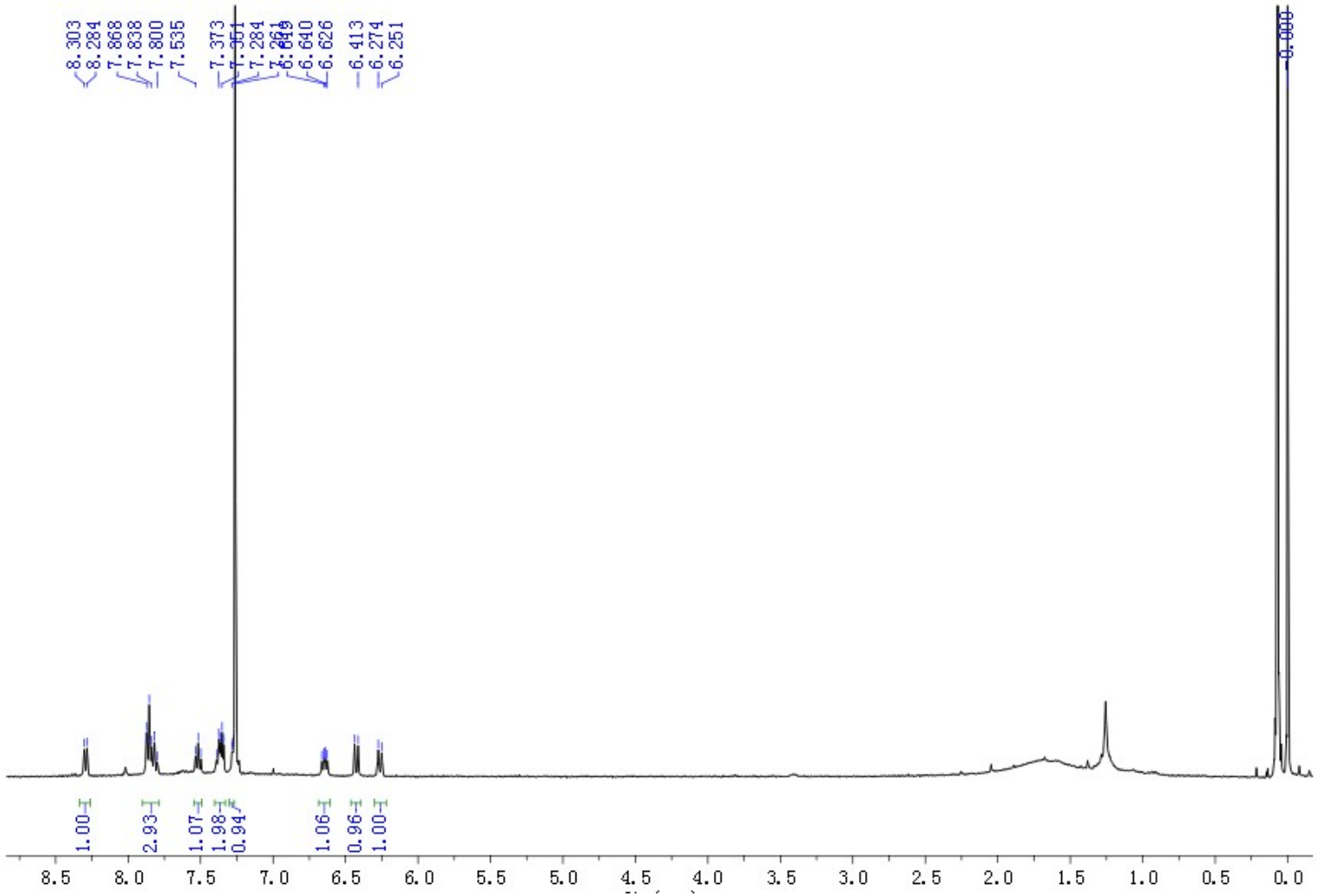
^{13}C NMR of 8-chloro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3b**)



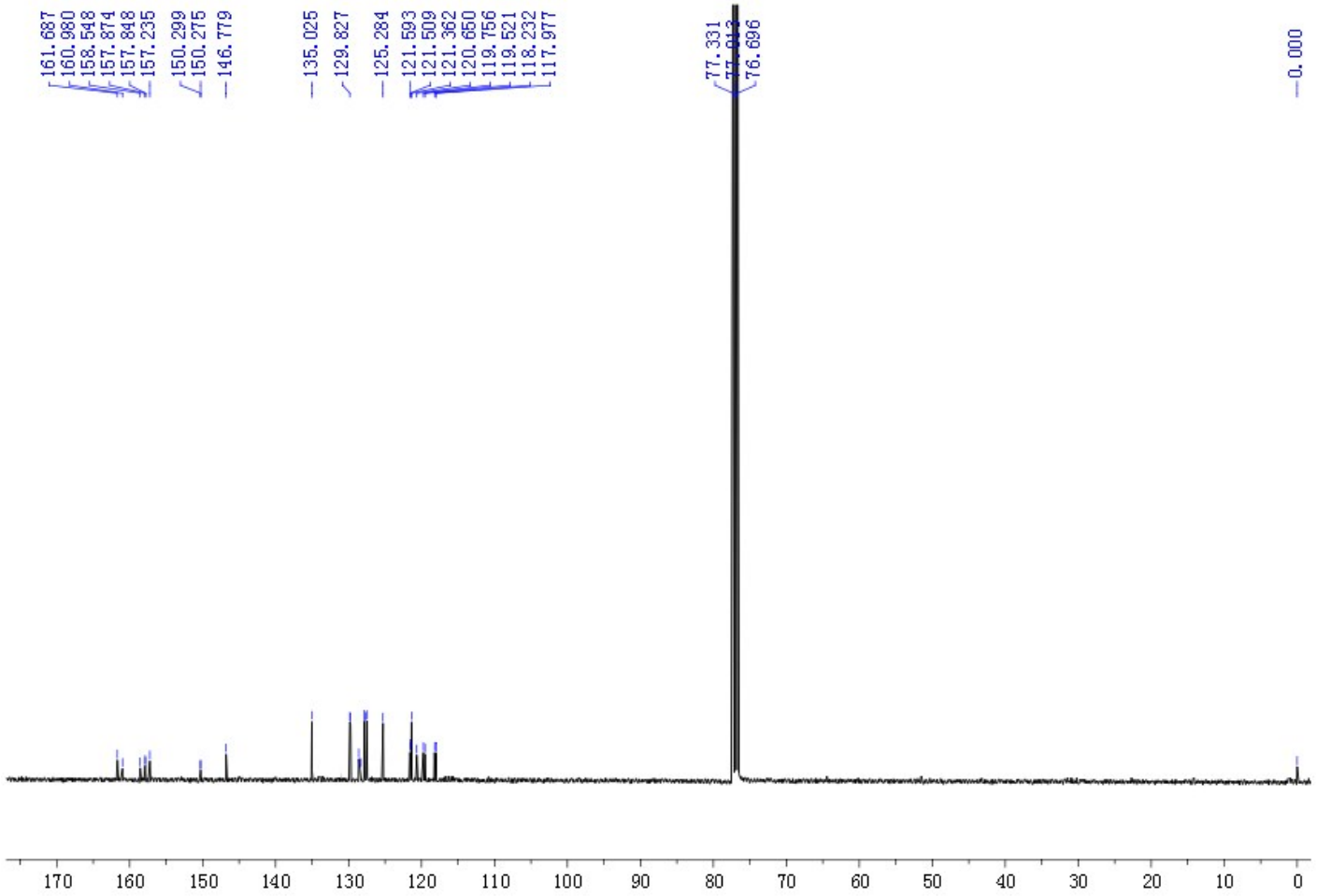
^{1}H NMR of 7-chloro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3c**)



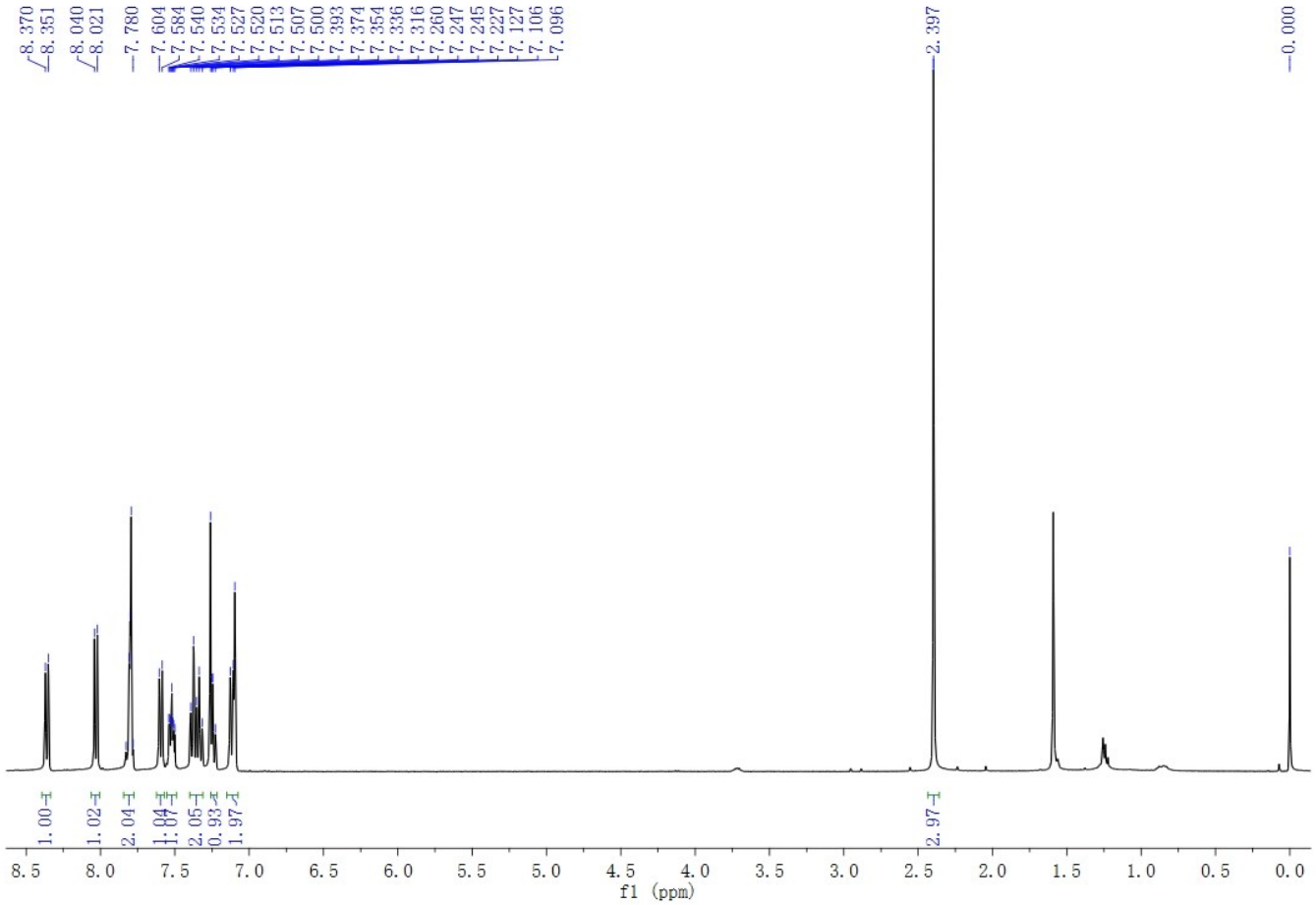
^{13}C NMR of 7-chloro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3c**)



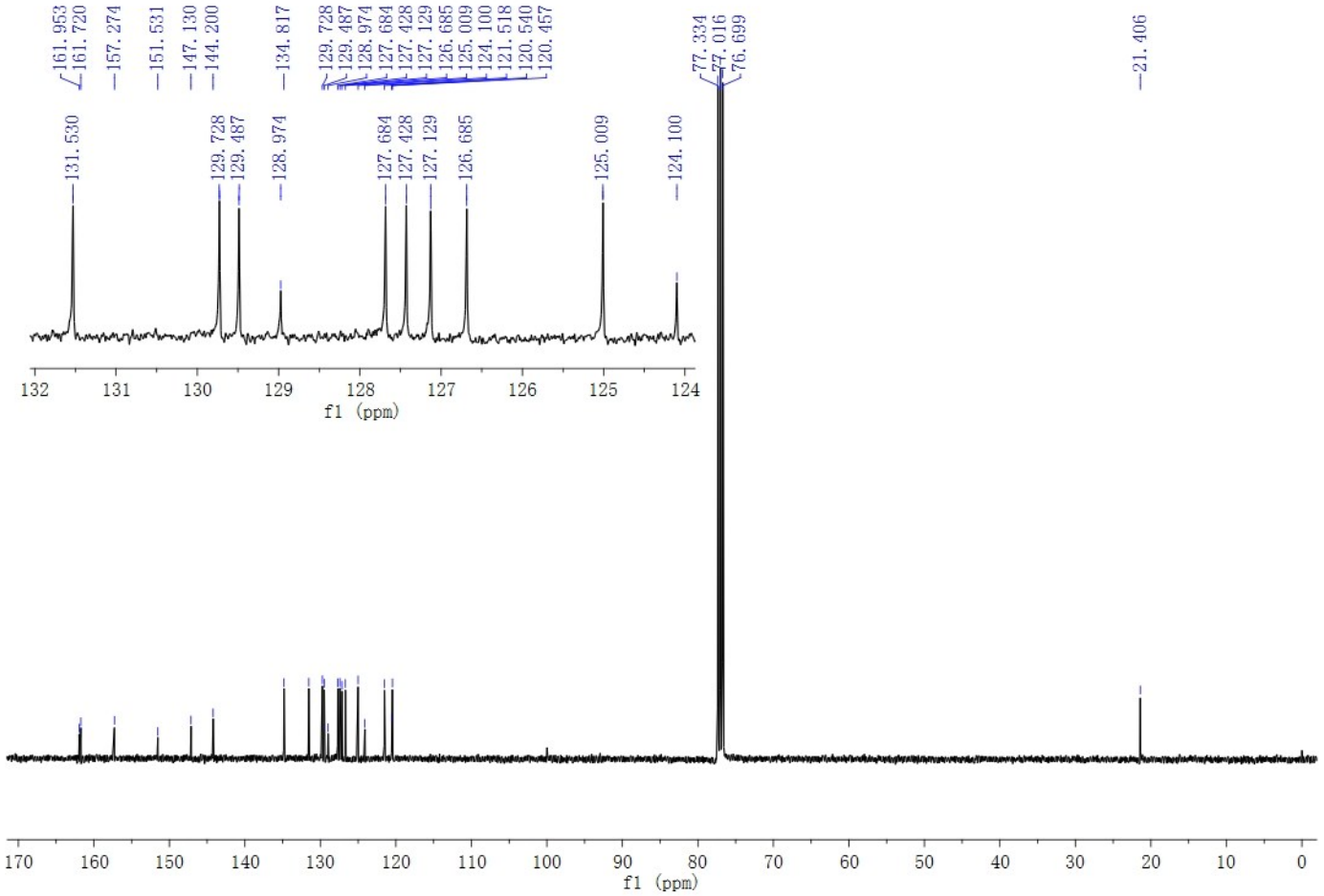
^{1}H NMR of 7-fluoro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3d**)



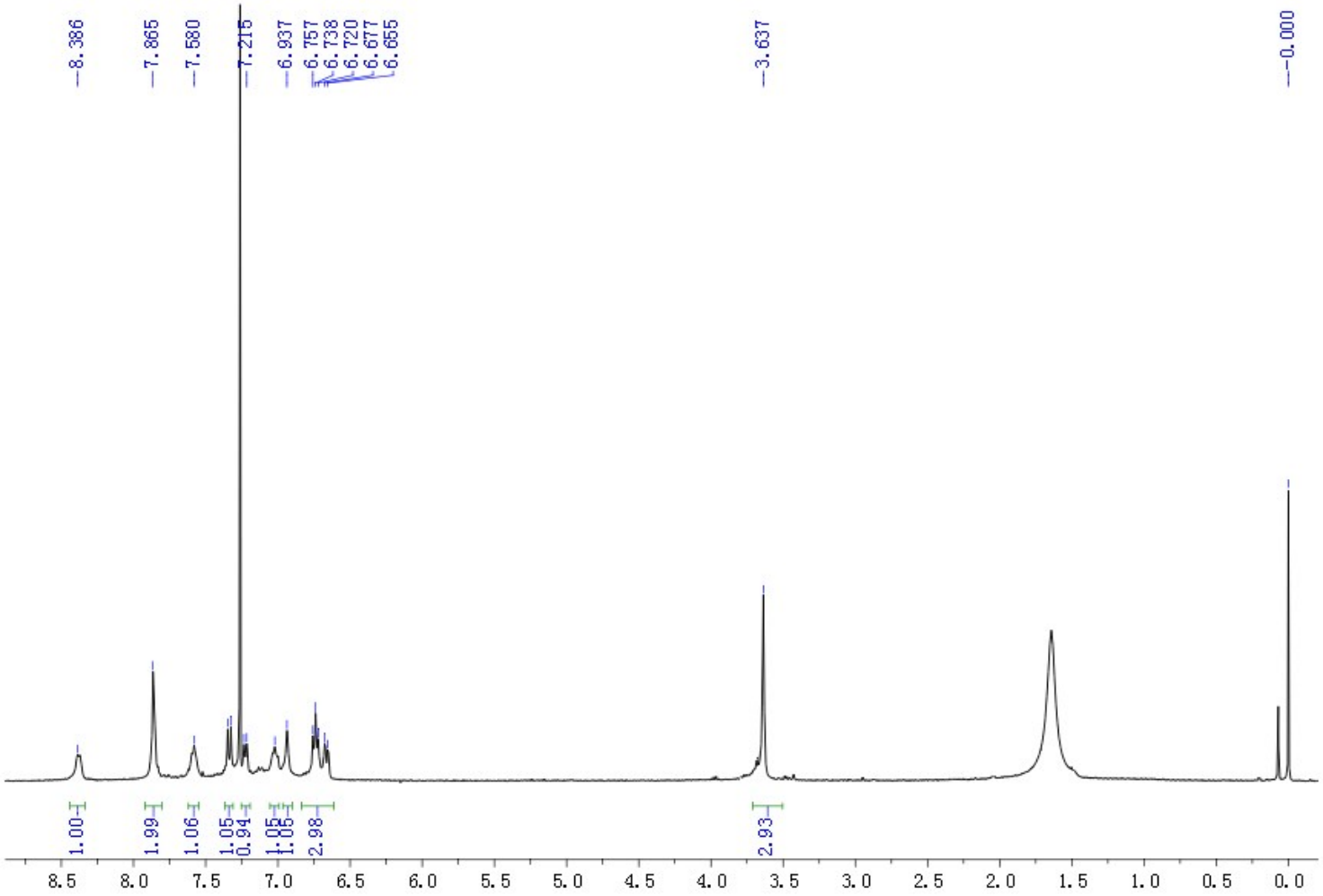
^{13}C NMR of 7-fluoro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3d**)



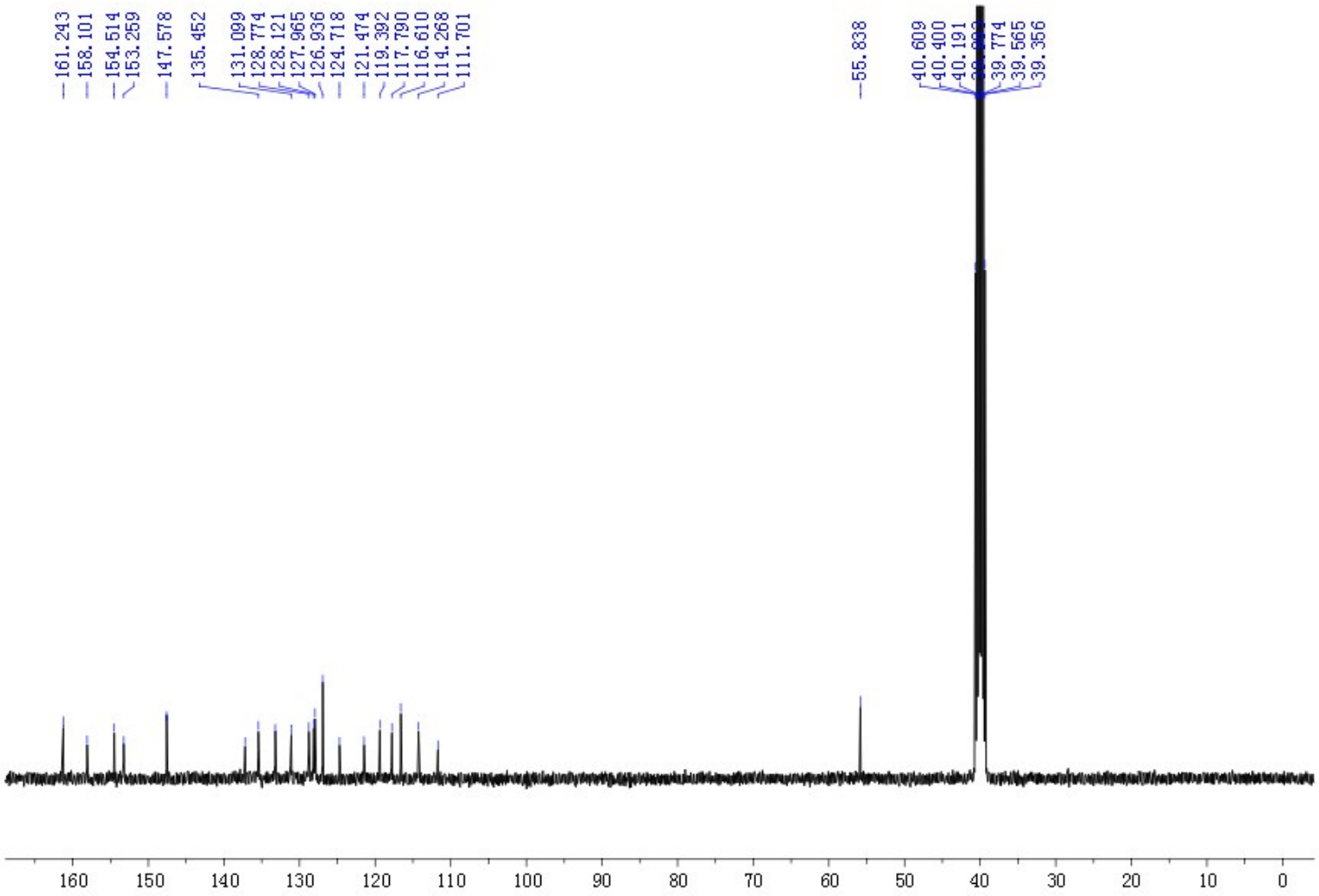
^{1}H NMR of 8-methyl-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3e**)



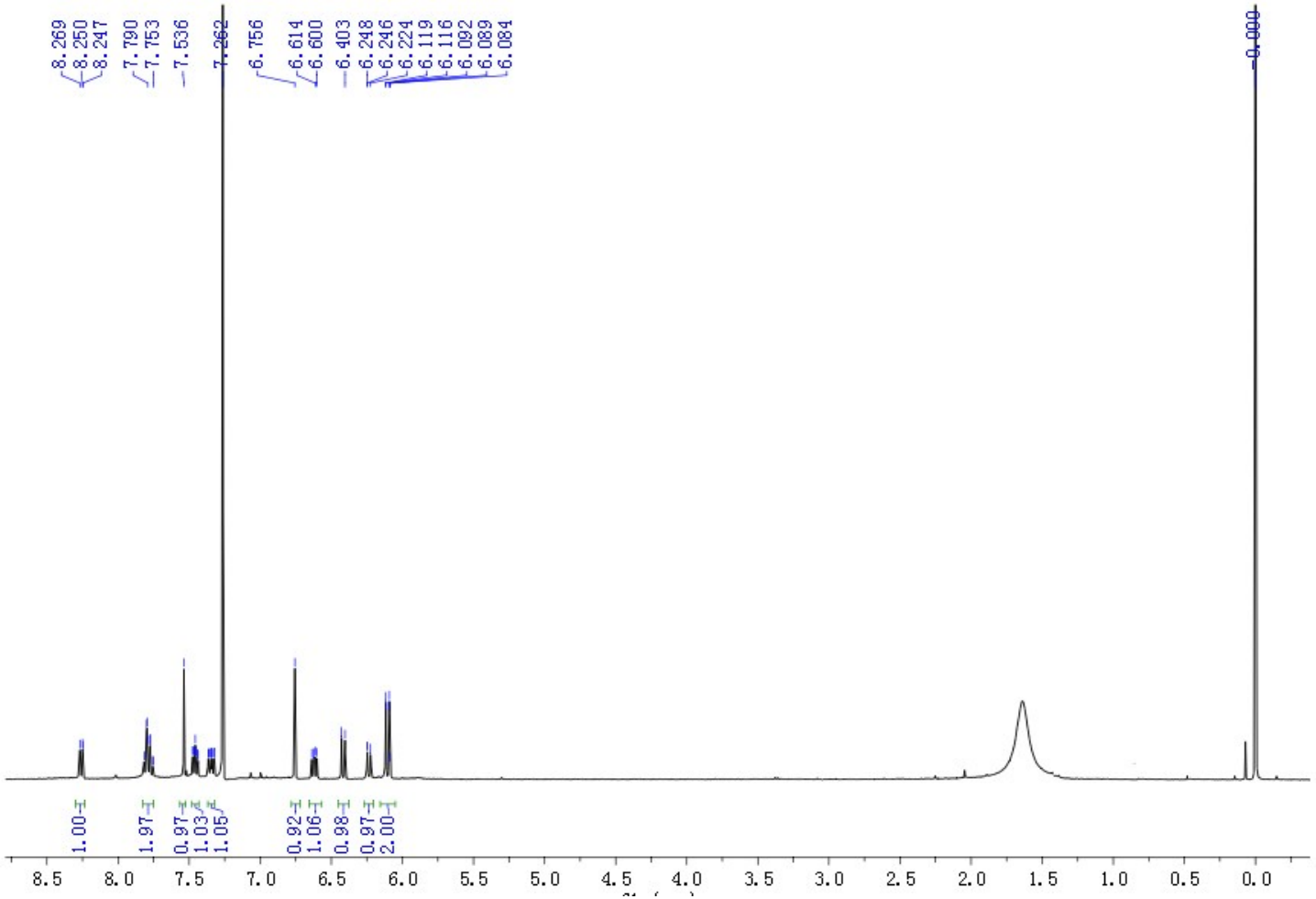
^{13}C NMR of 8-methyl-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3e**)



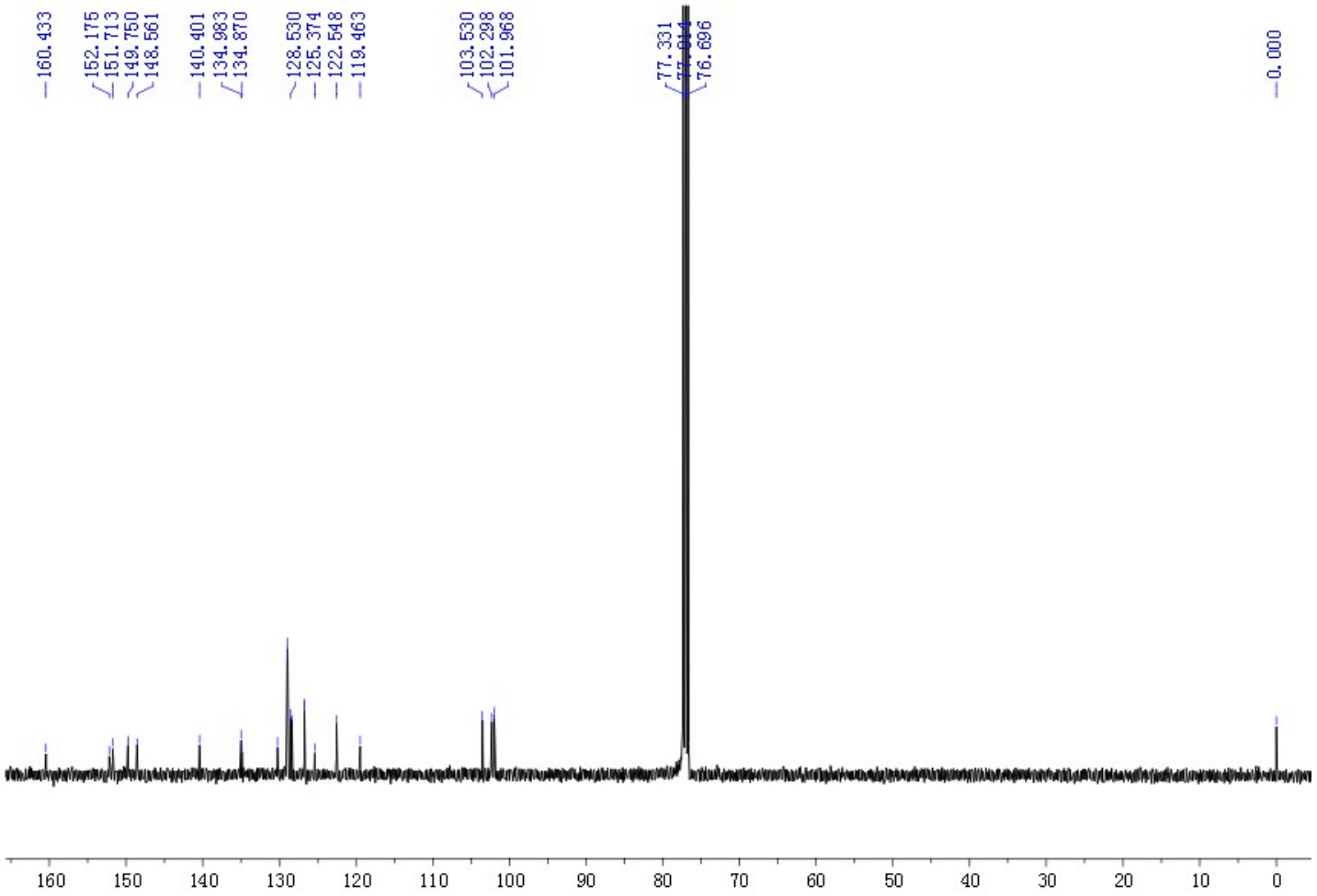
^{1}H NMR of 7-methoxy-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3f**)



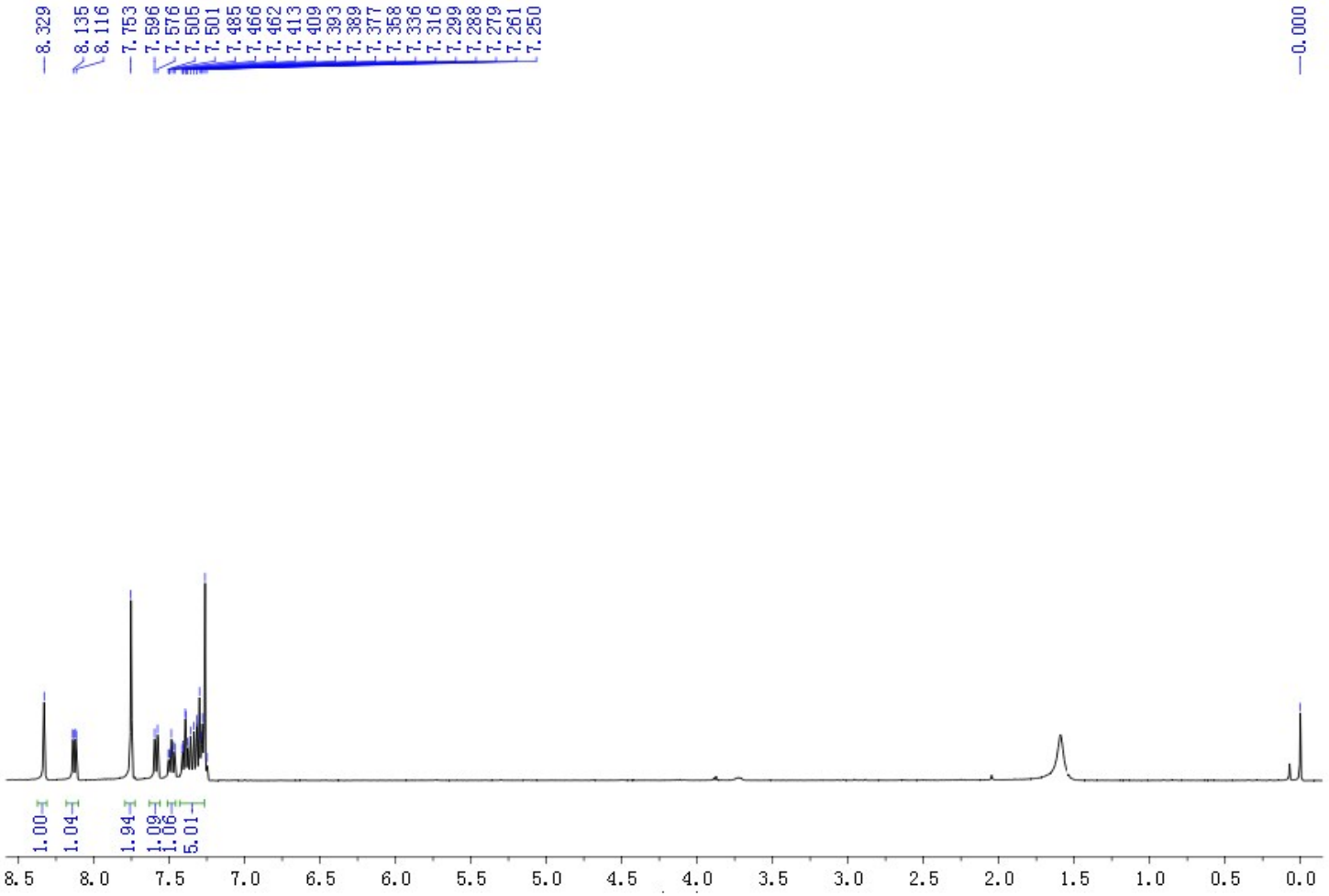
^{13}C NMR of 7-methoxy-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3f**)



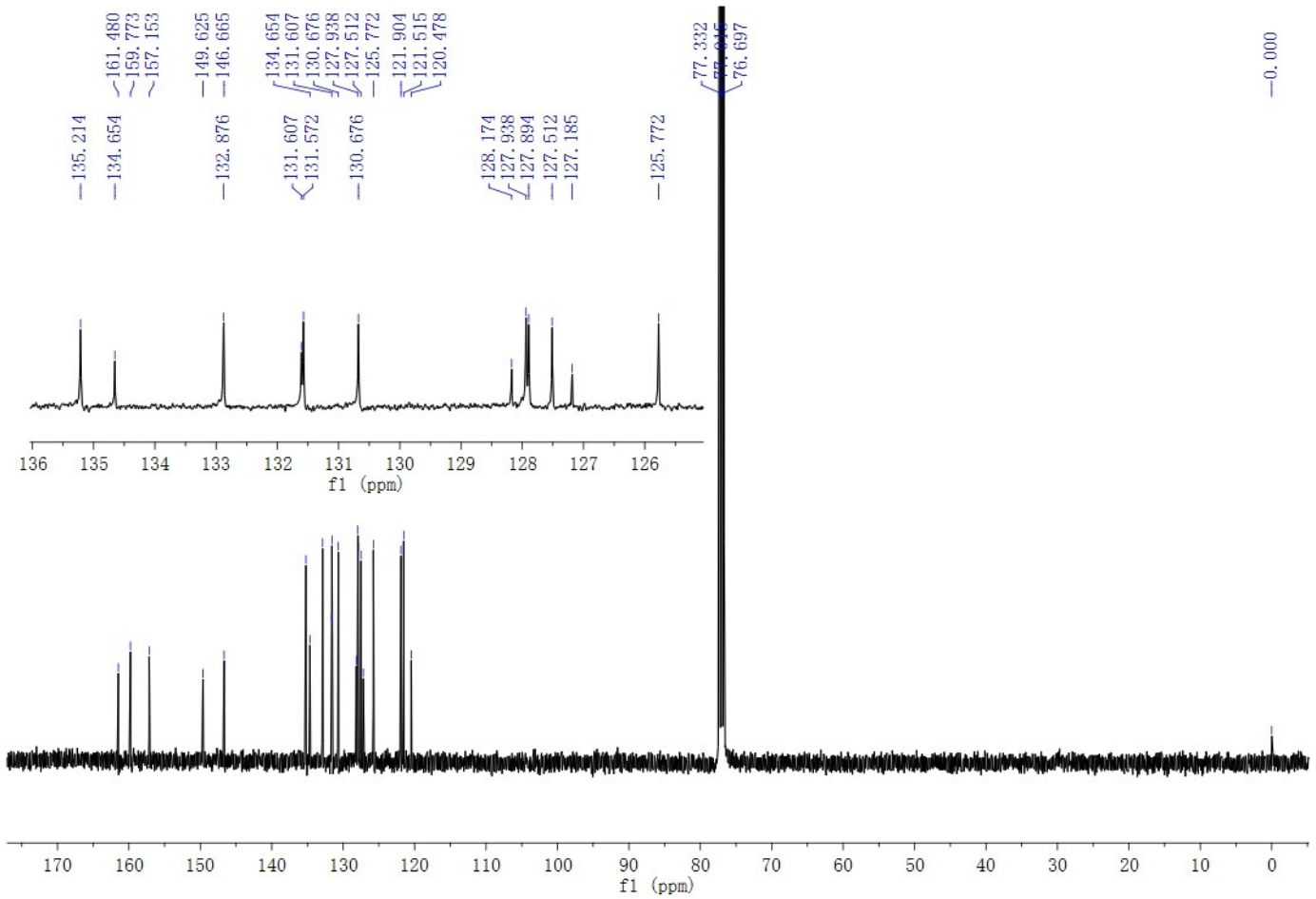
^{1}H NMR of 5*H*-[1,3]dioxolo[4",5":4',5']benzo[1',2':6,7]benzo[2,3][1,4]oxazepino[5,4-*b*]quinazolin-5-one (**3g**)



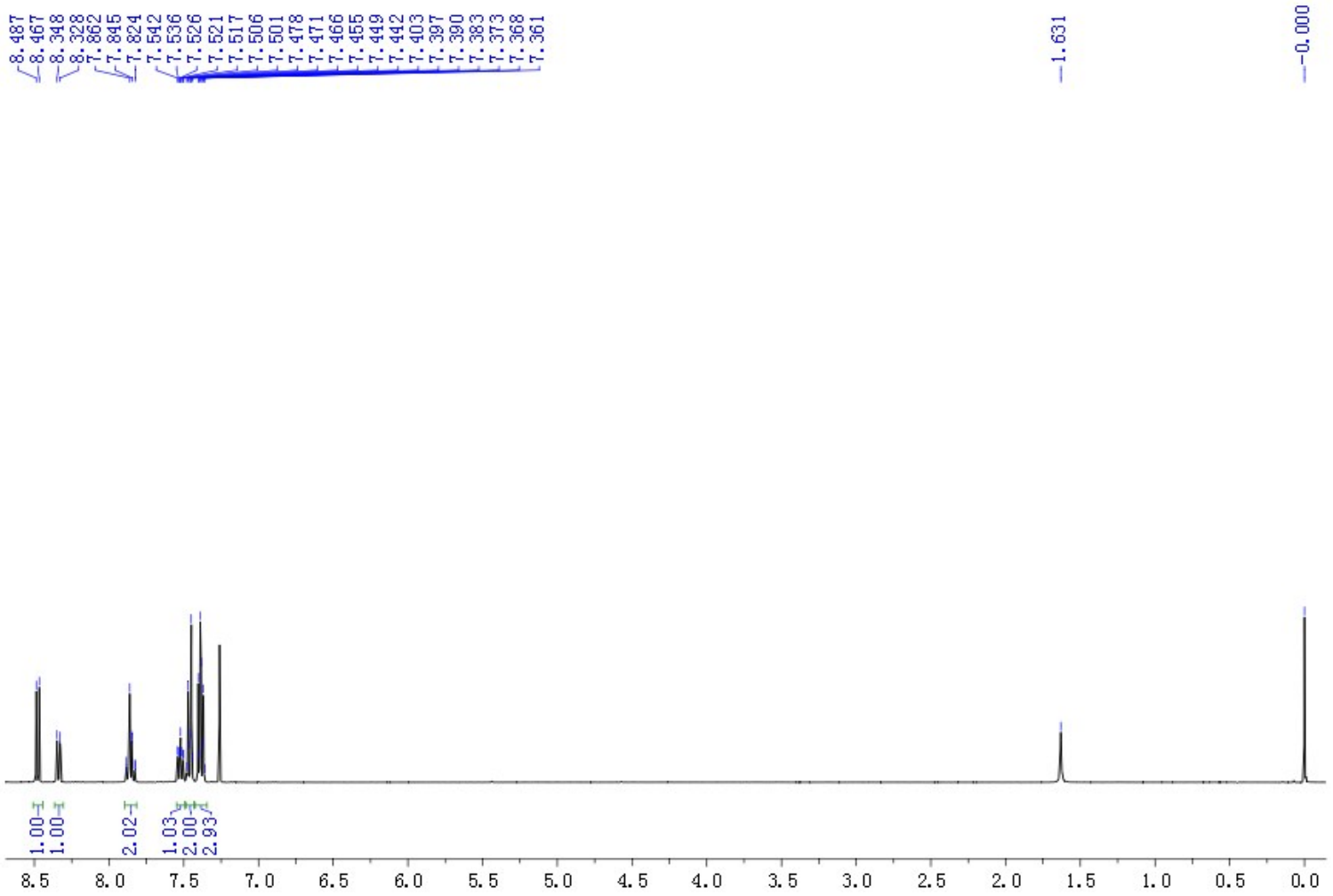
^{13}C NMR of 5*H*-[1,3]dioxolo[4",5":4',5']benzo[1',2':6,7]benzo[2,3][1,4]oxazepino[5,4-*b*]quinazolin-5-one (**3g**)



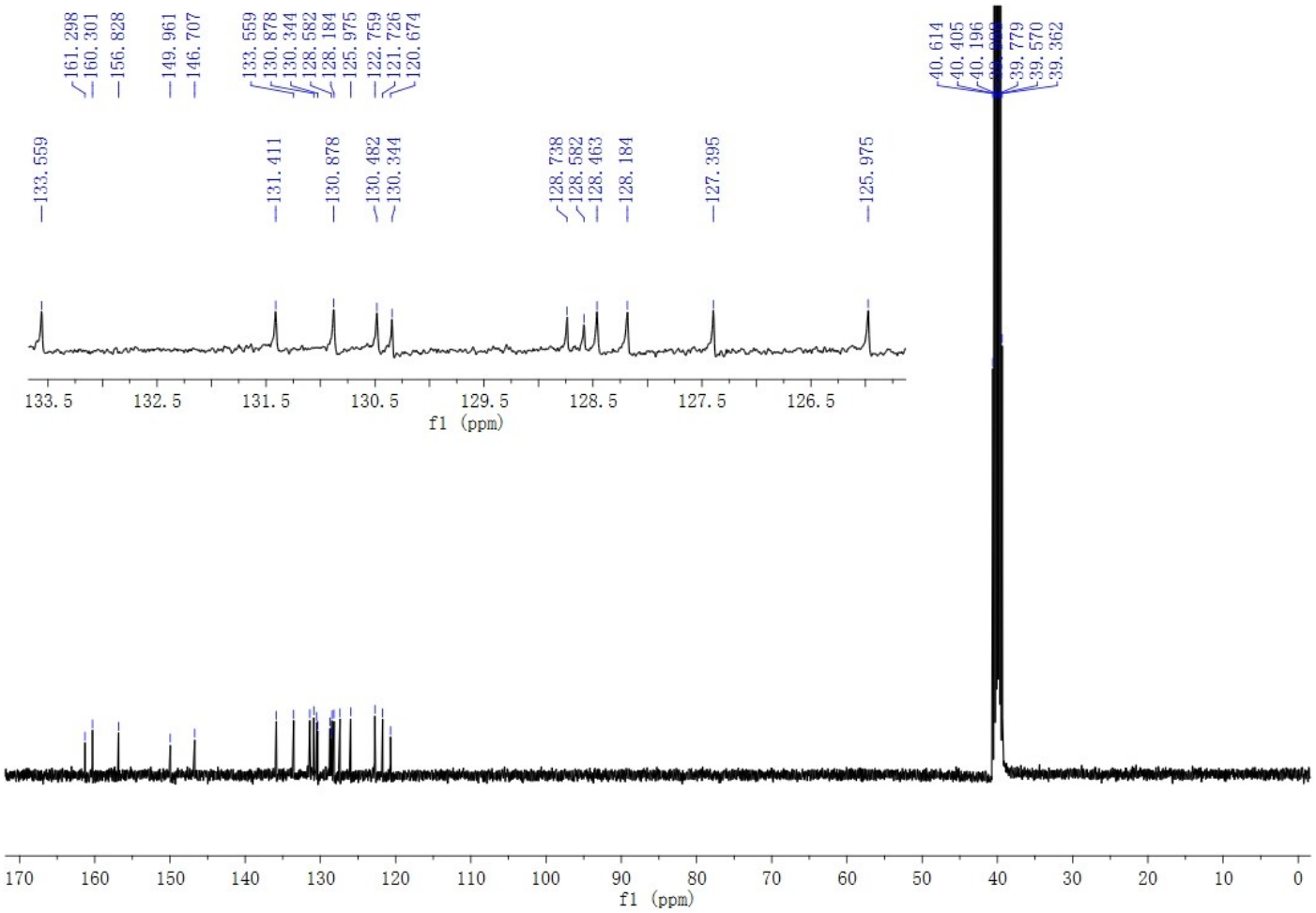
^{1}H NMR of 2-chloro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3h**)



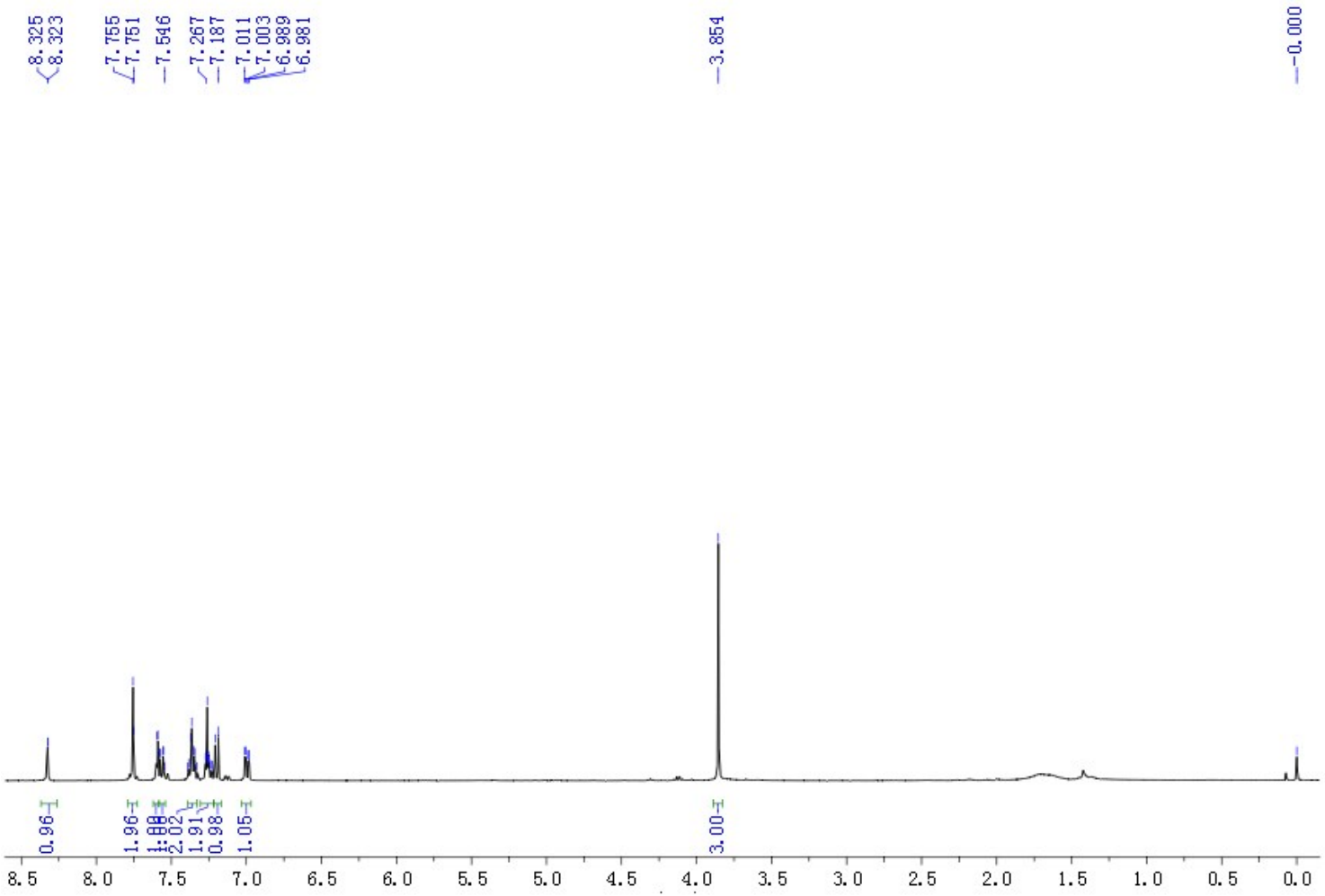
^{13}C NMR of 2-chloro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3h**)



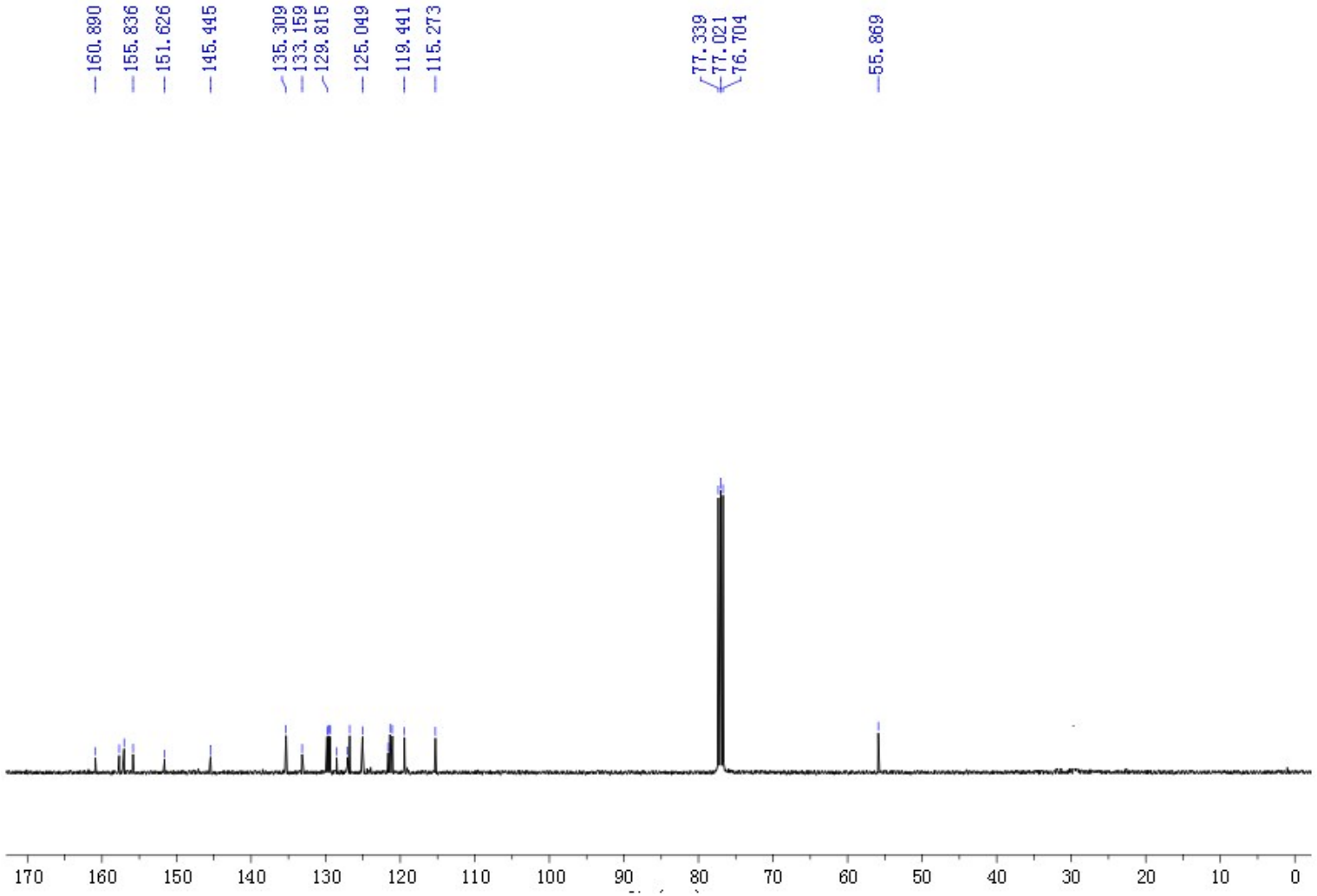
^{1}H NMR of 2,7-dichloro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3i**)



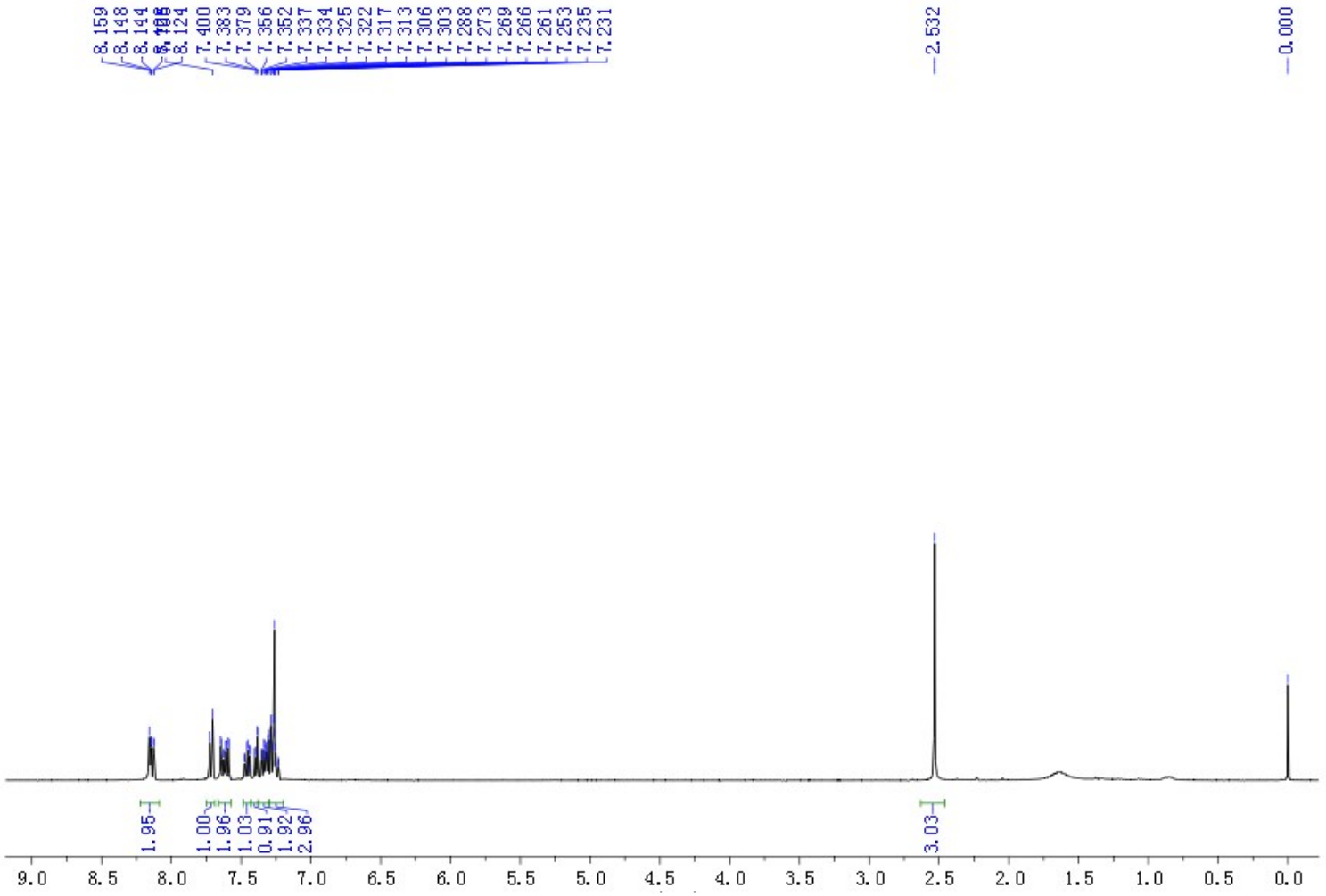
^{13}C NMR of 2,7-dichloro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3i**)



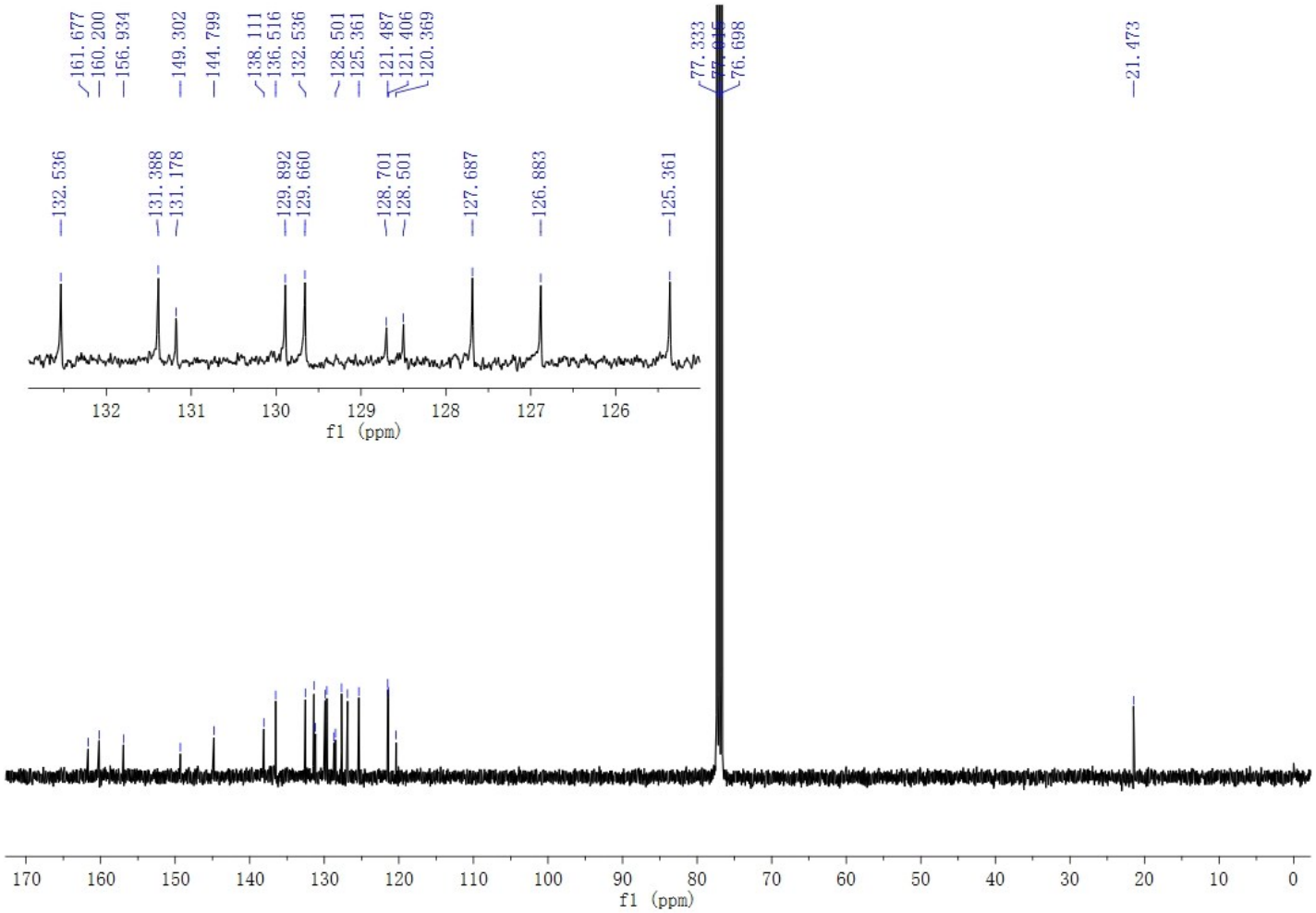
^{1}H NMR of 2-chloro-7-methoxy-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3j**)



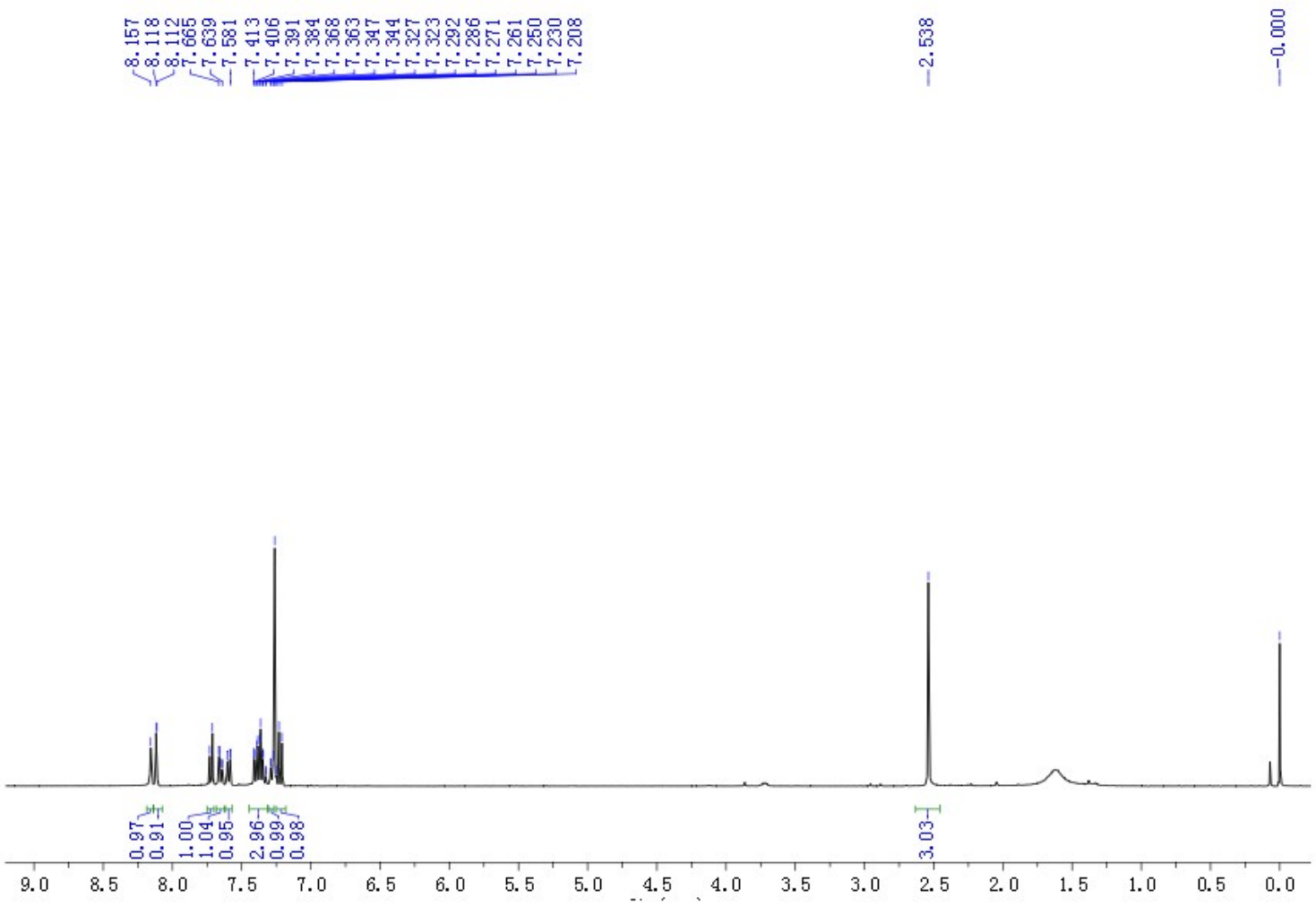
^{13}C NMR of 2-chloro-7-methoxy-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3j**)



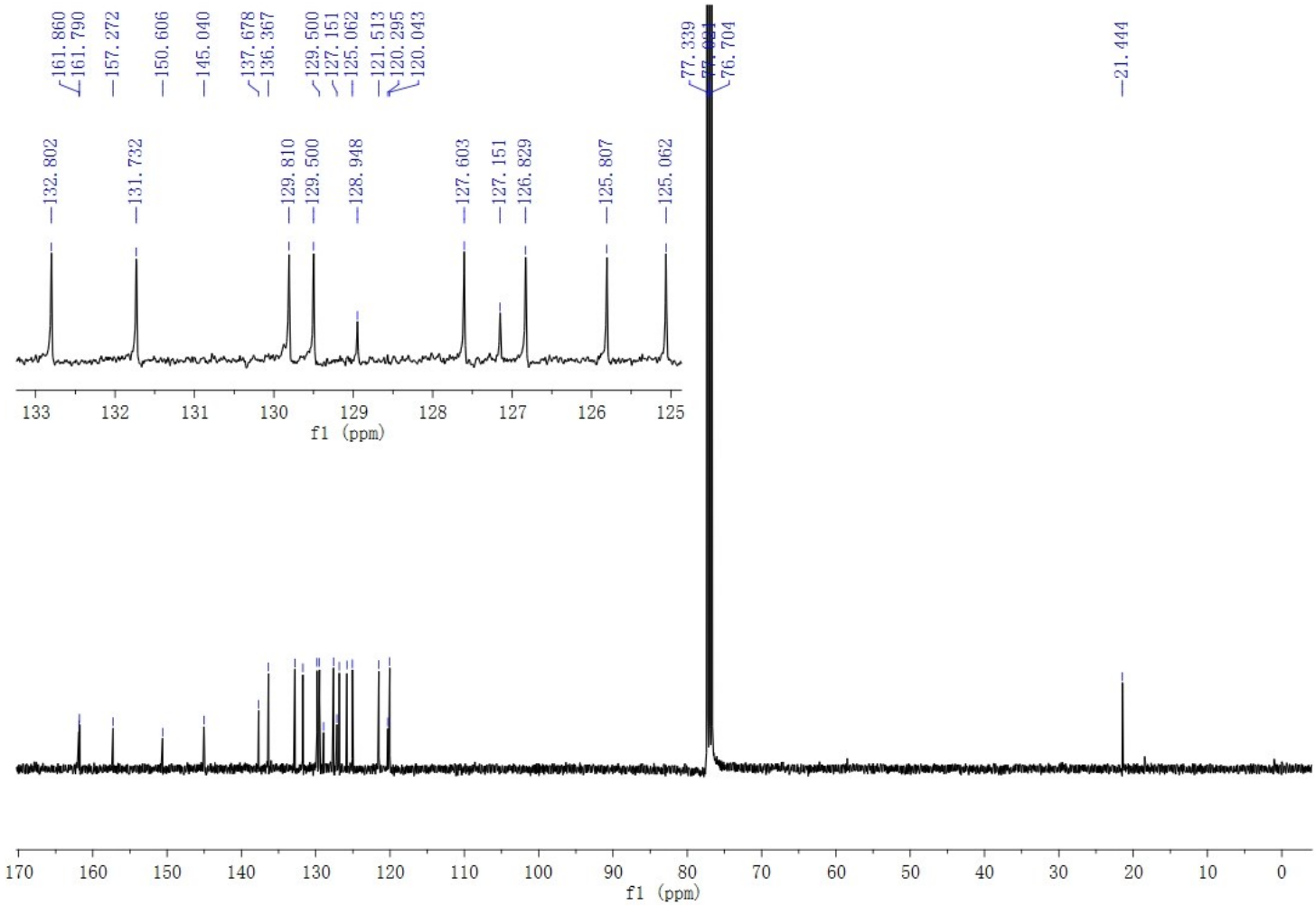
^{1}H NMR of 2-methyl-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3k**)



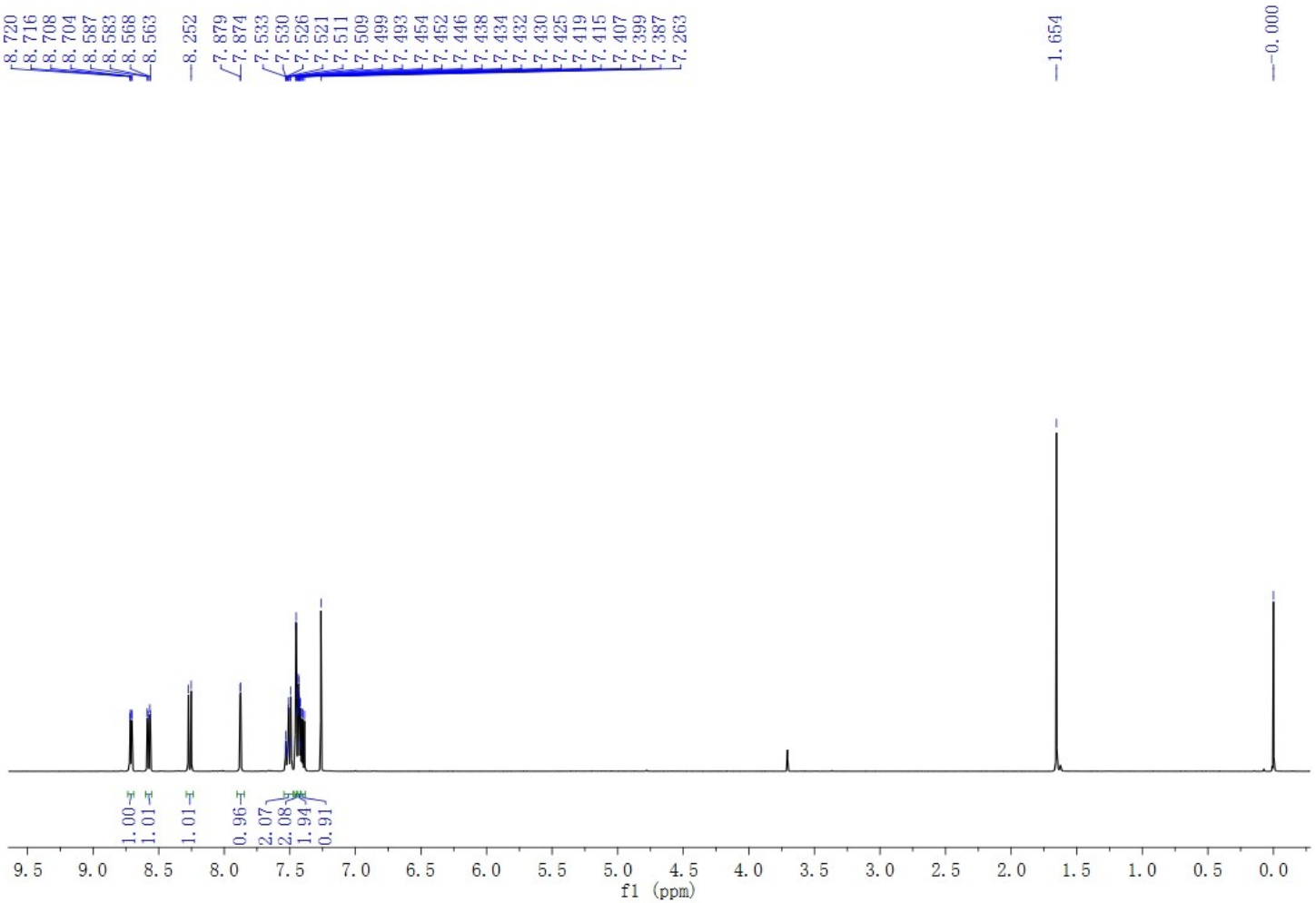
^{13}C NMR of 2-methyl-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3k**)



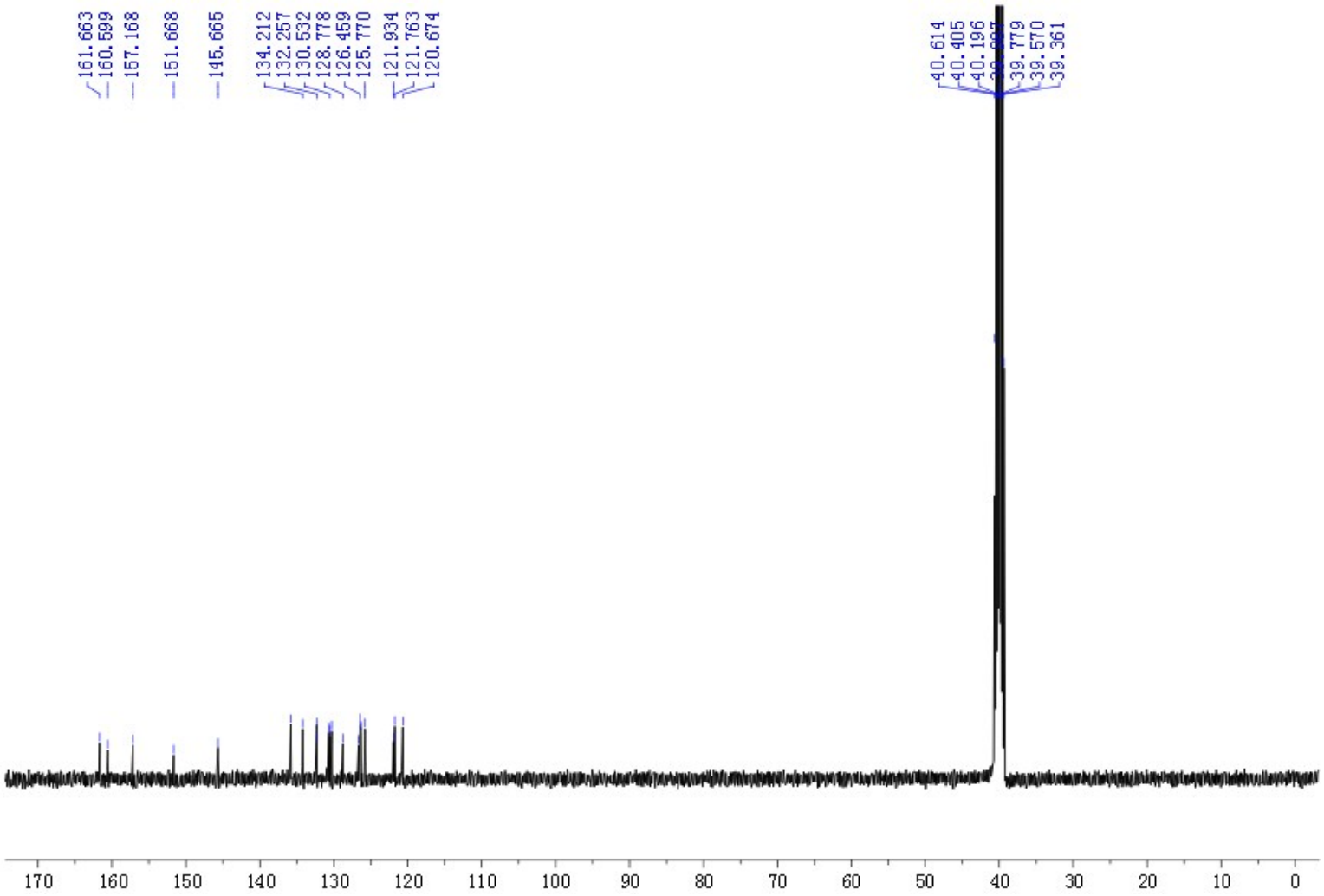
^{1}H NMR of 7-chloro-2-methyl-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3l**)



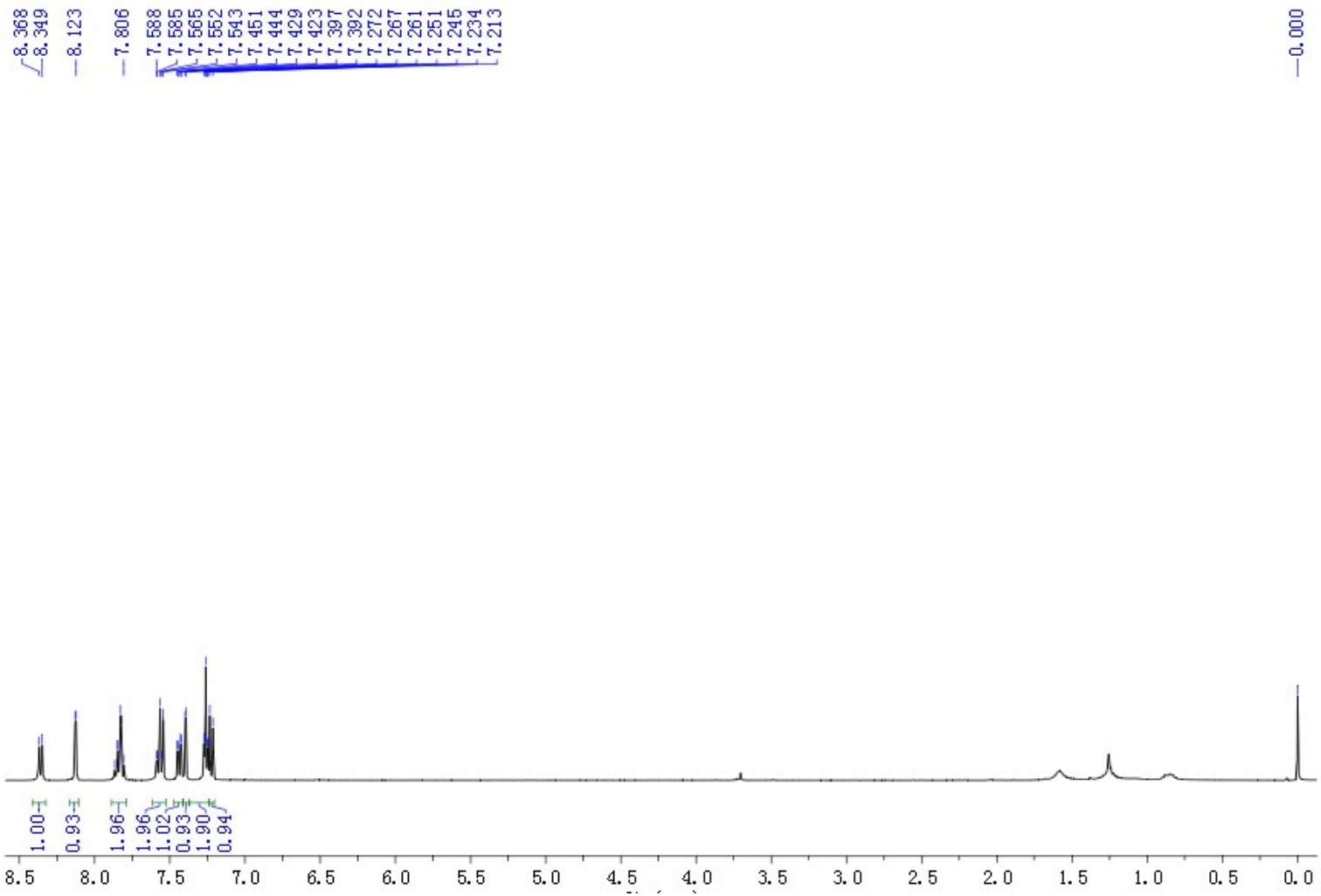
^{13}C NMR of 7-chloro-2-methyl-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3l**)



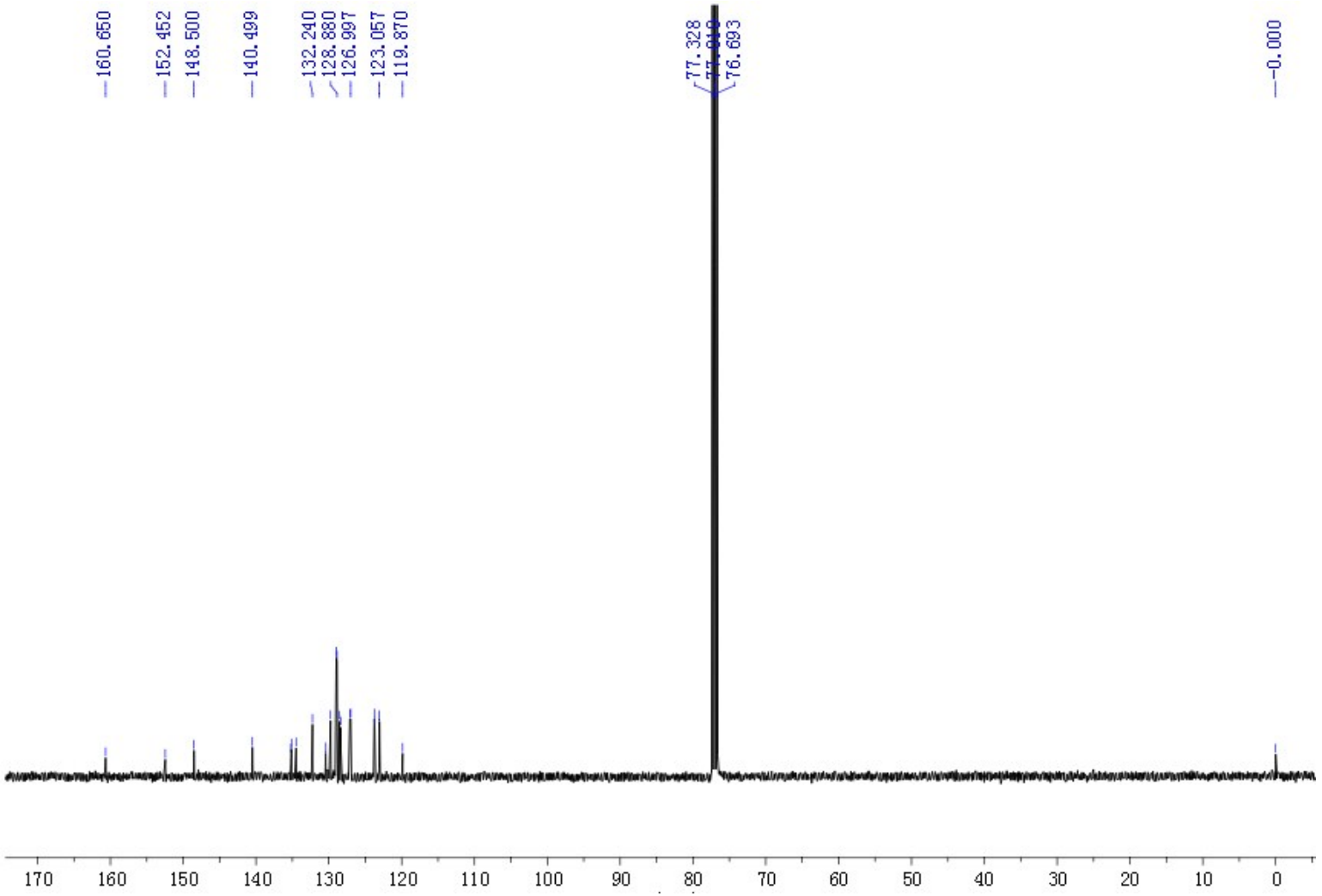
^{1}H NMR of 12-chloro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3m**)



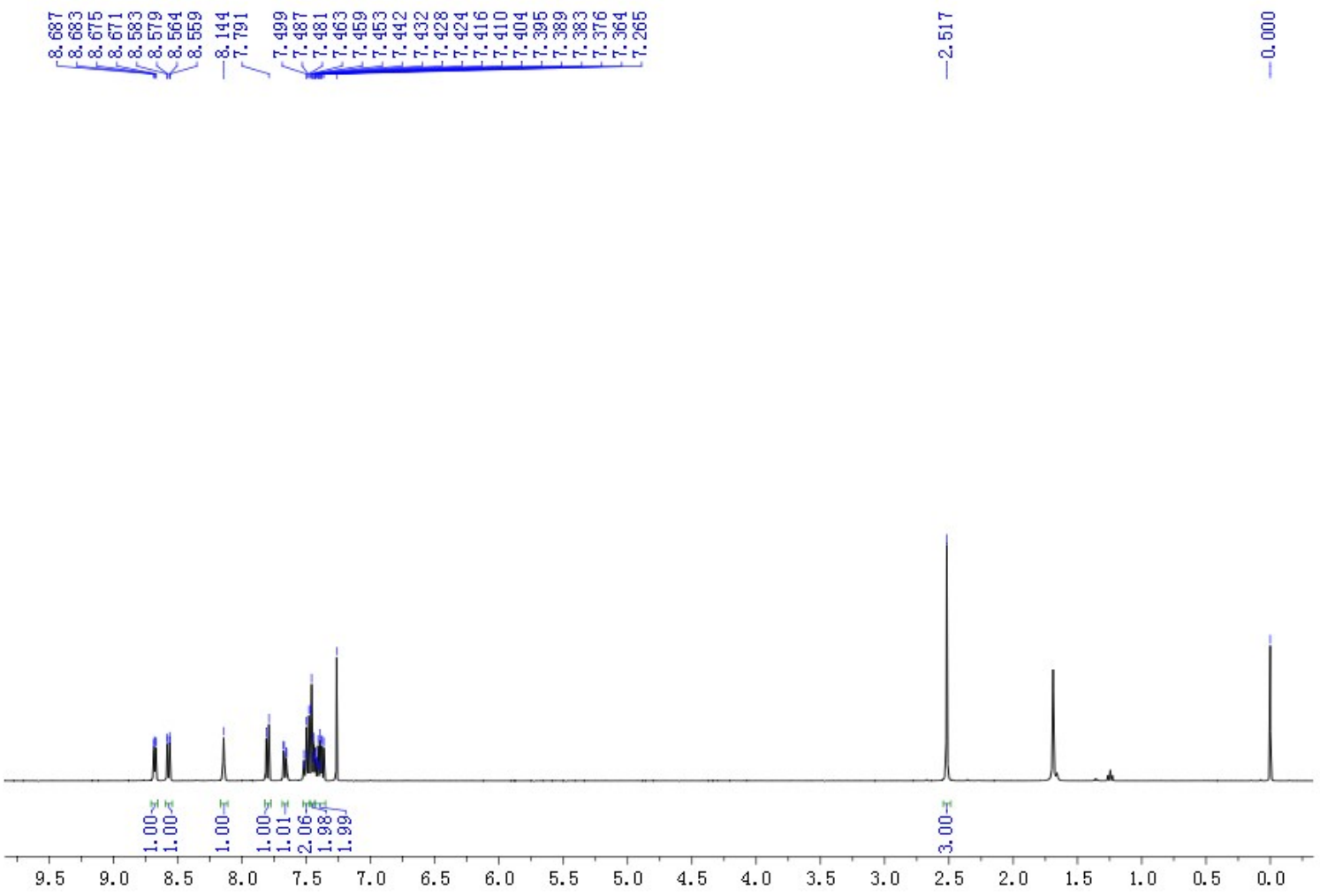
^{13}C NMR of 12-chloro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3m**)



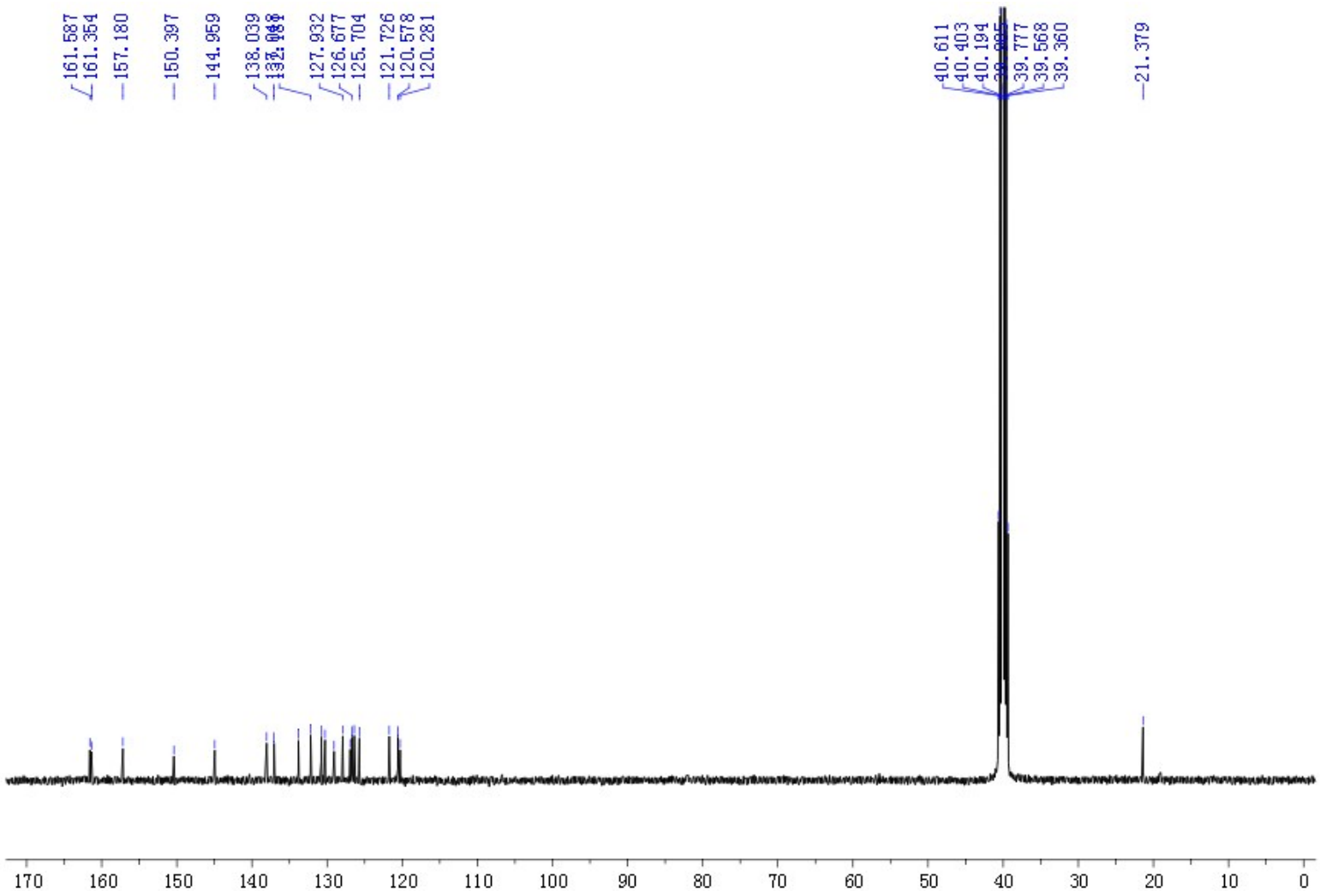
^{1}H NMR of 7,12-dichloro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3n**)



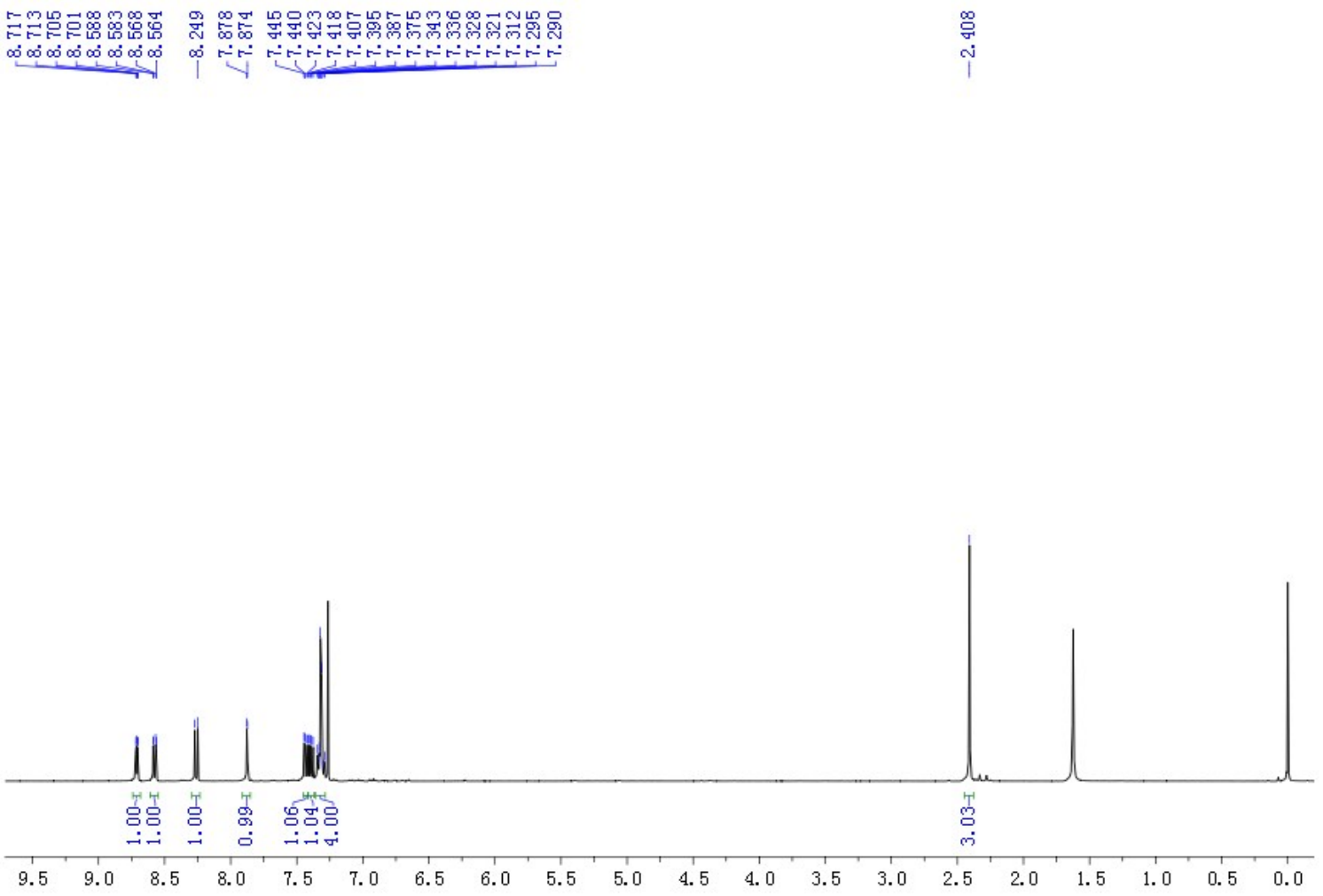
^{13}C NMR of 7,12-dichloro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3n**)



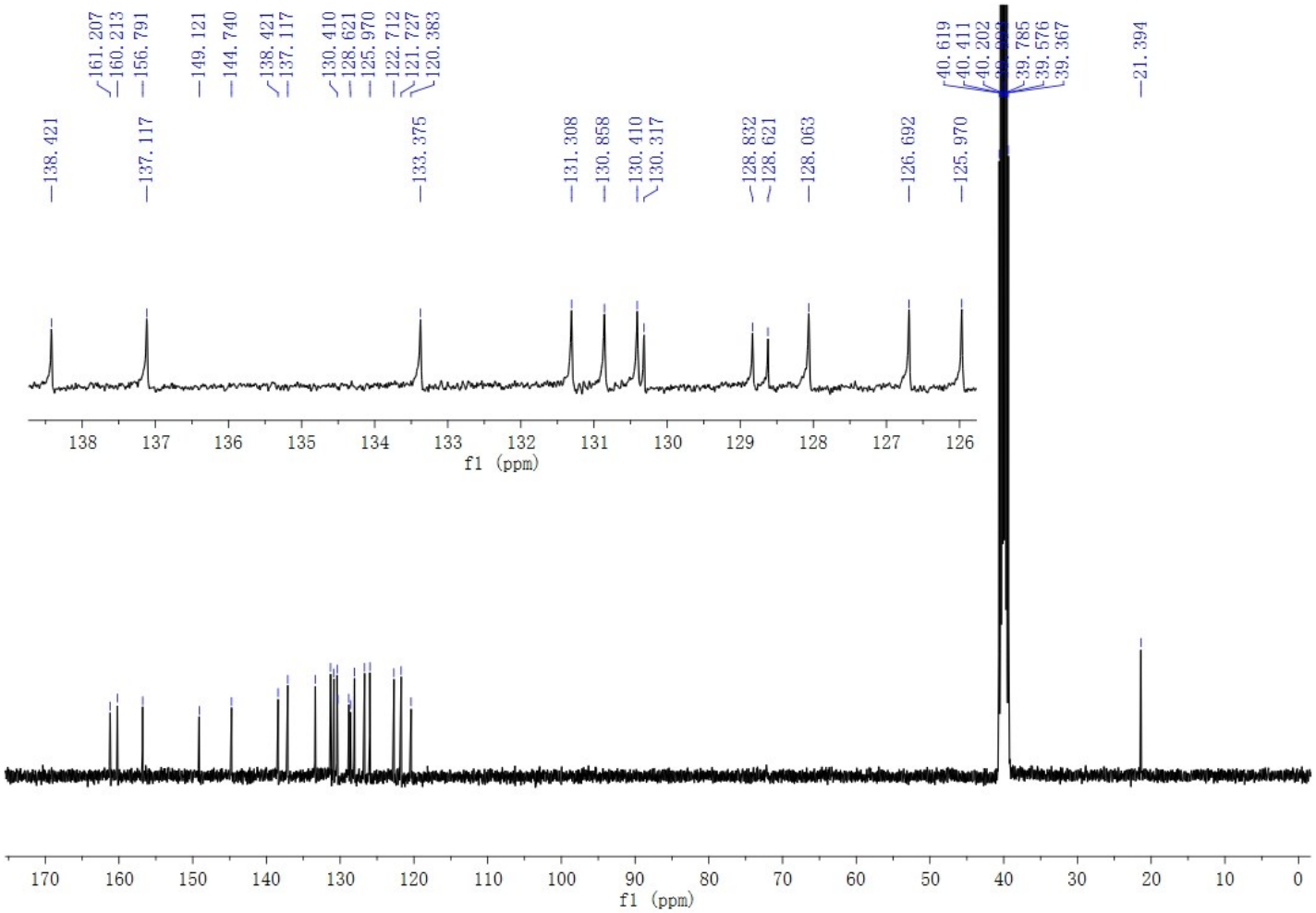
^{1}H NMR of 12-methyl-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3o**)



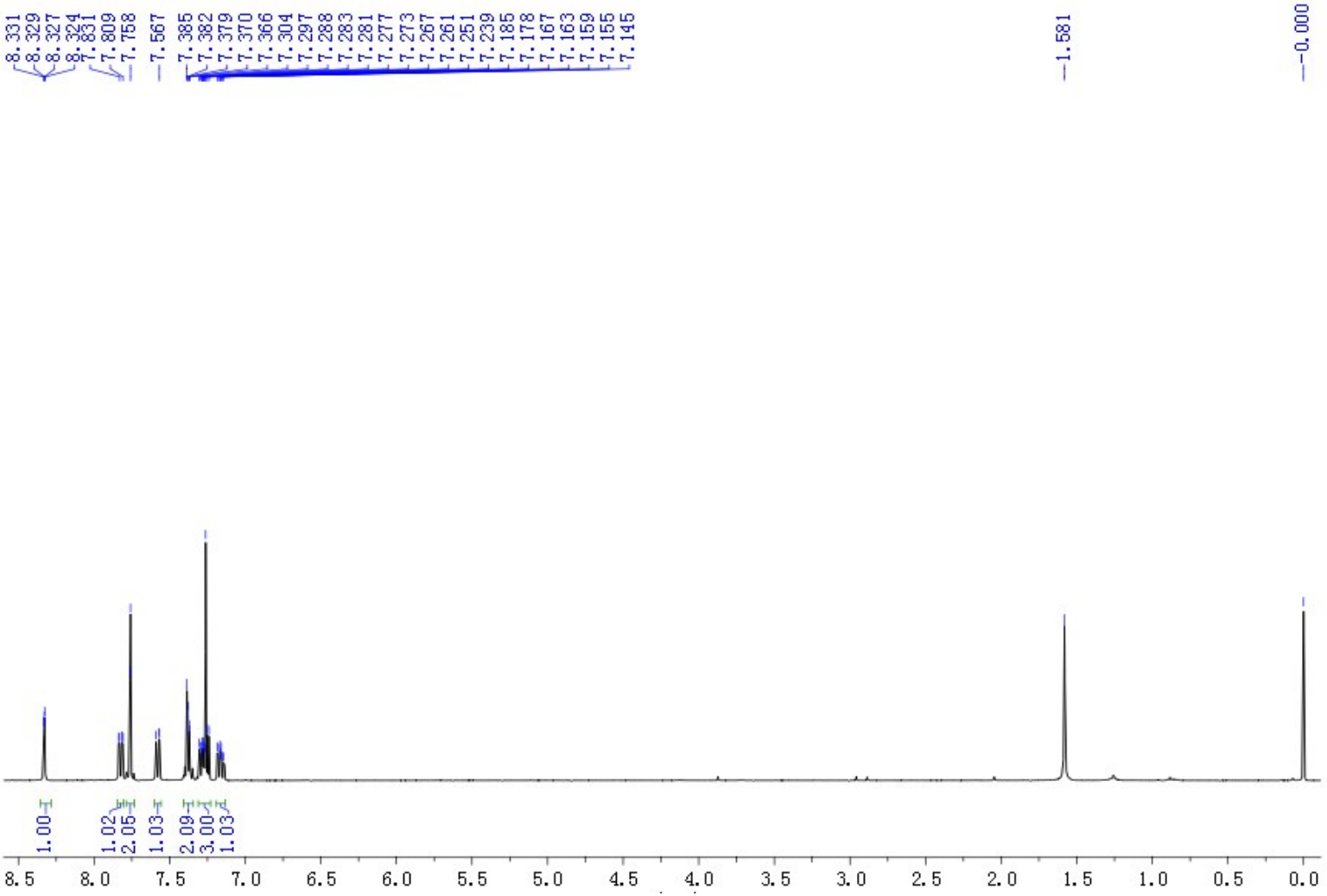
^{13}C NMR of 12-methyl-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3o**)



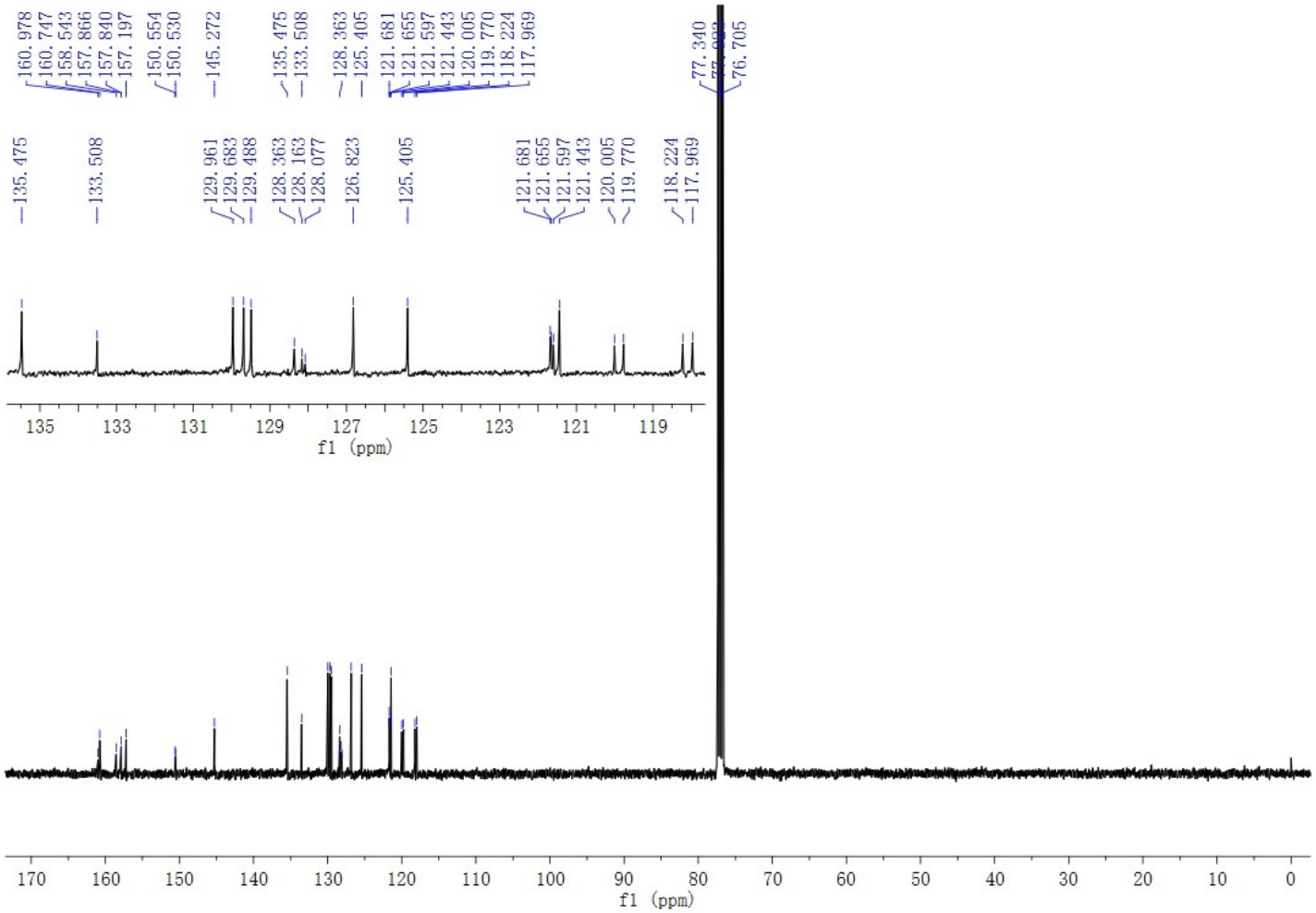
^{1}H NMR of 7-chloro-12-methyl-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3p**)



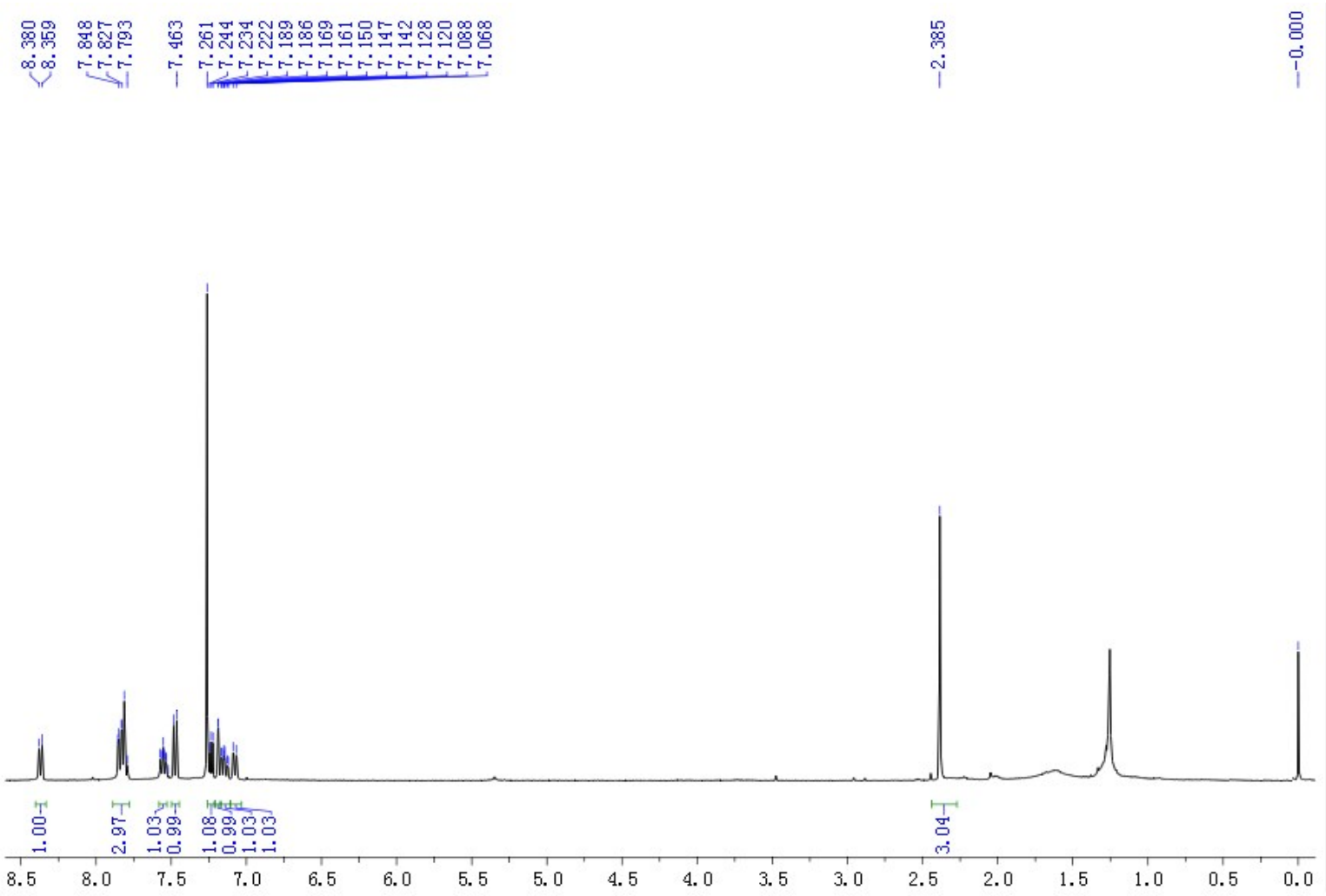
^{13}C NMR of 7-chloro-12-methyl-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3p**)



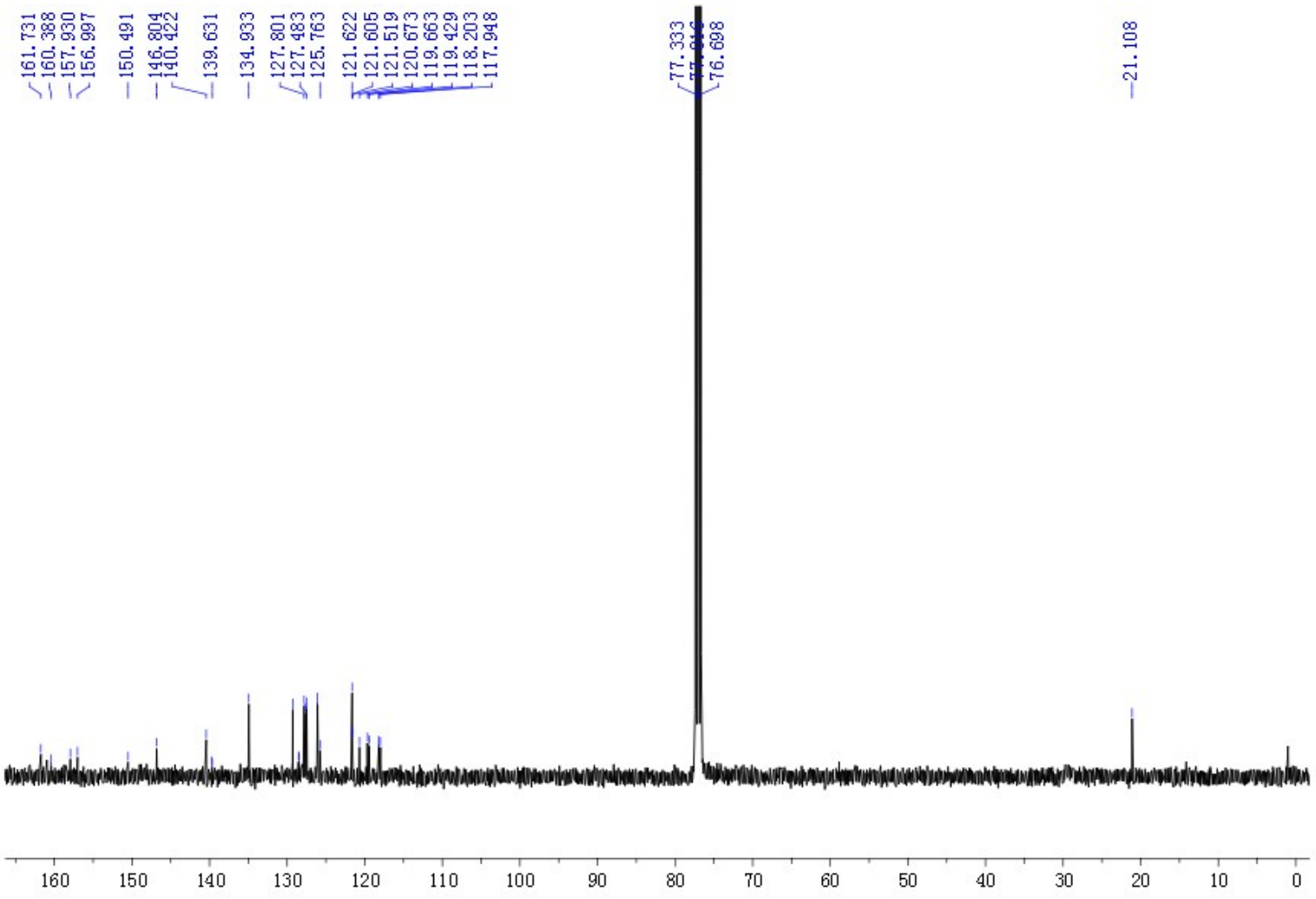
^{1}H NMR of 2-chloro-7-fluoro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3q**)



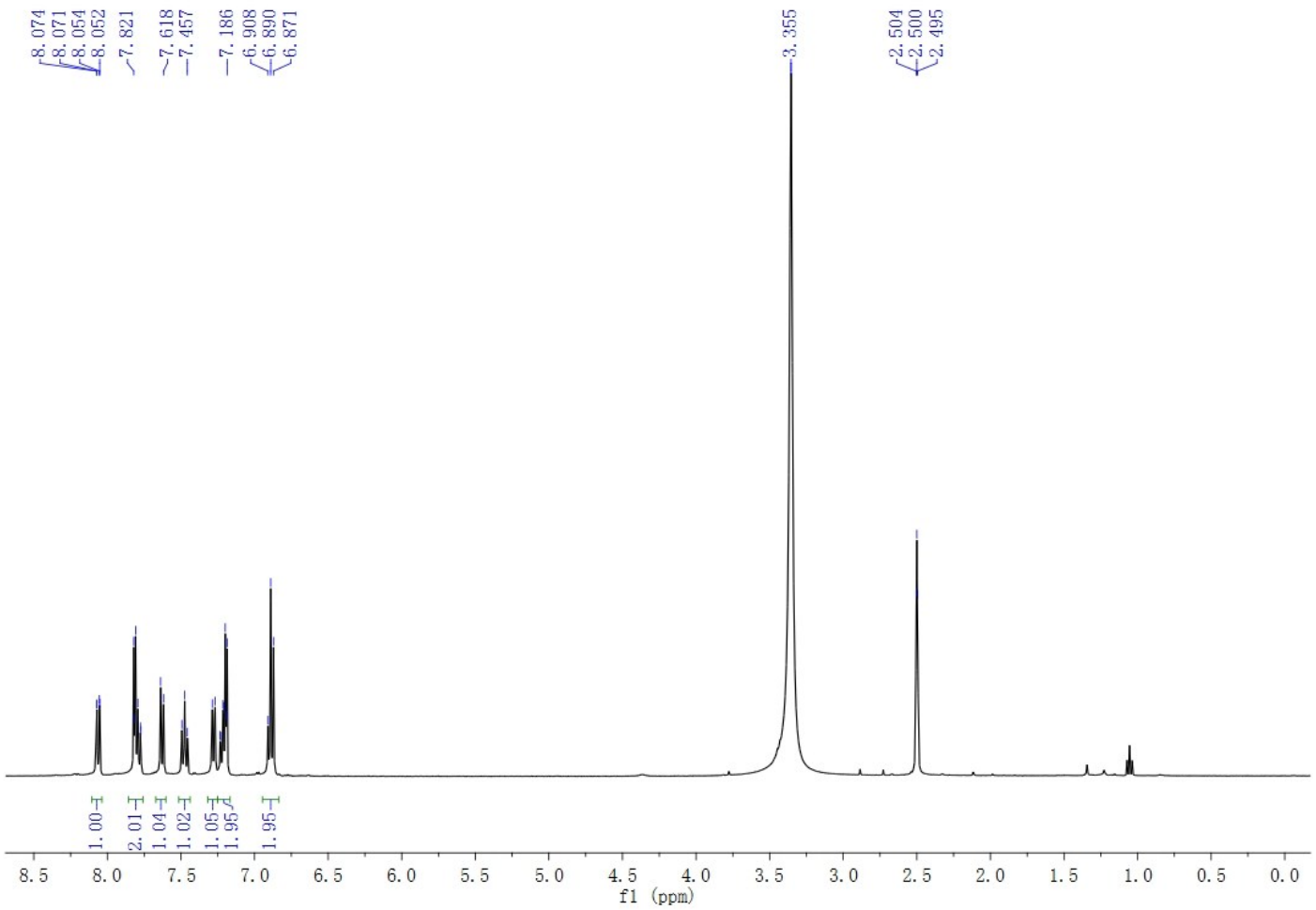
^{13}C NMR of 2-chloro-7-fluoro-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3q**)



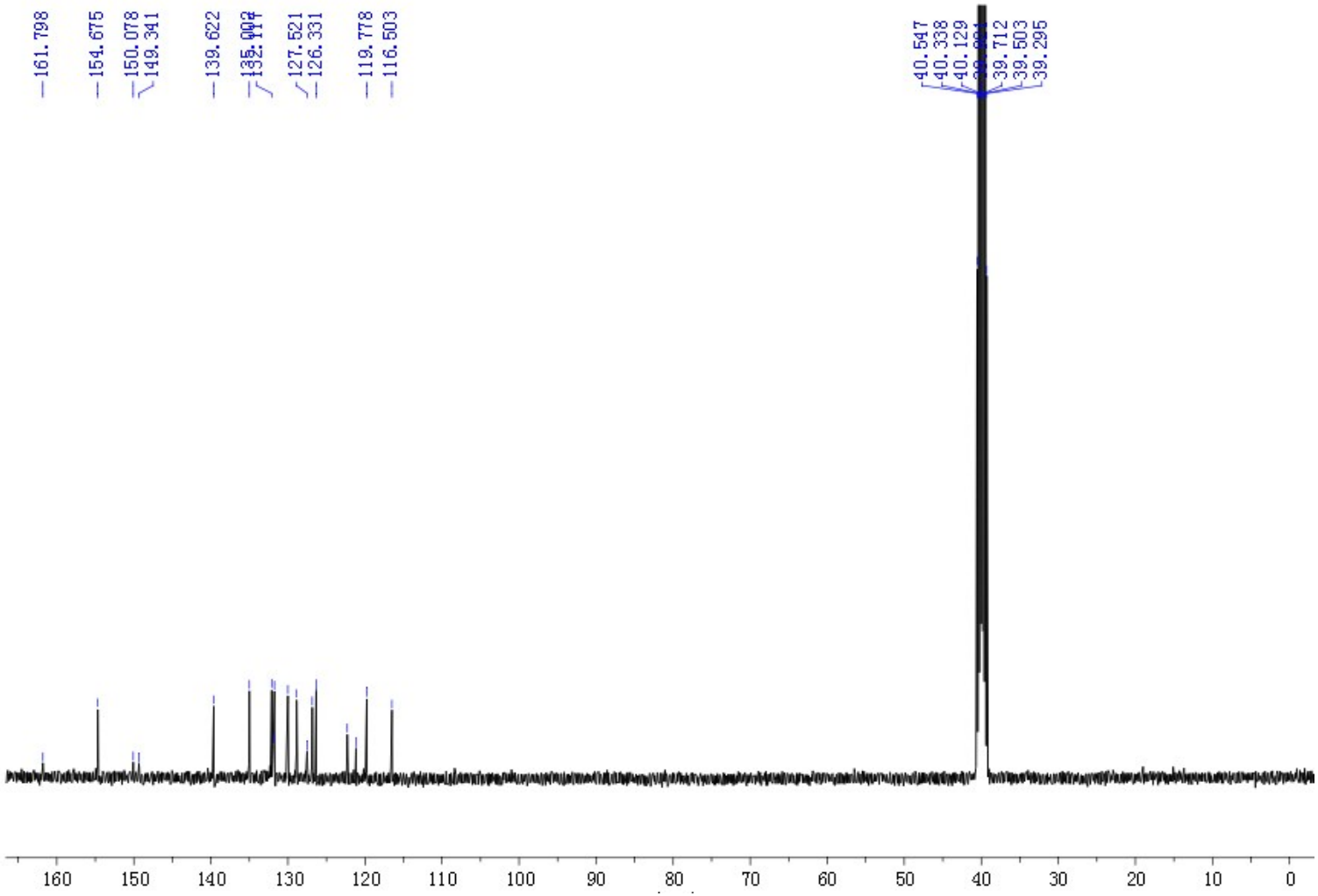
^{1}H NMR of 7-fluoro-2-methyl-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3r**)



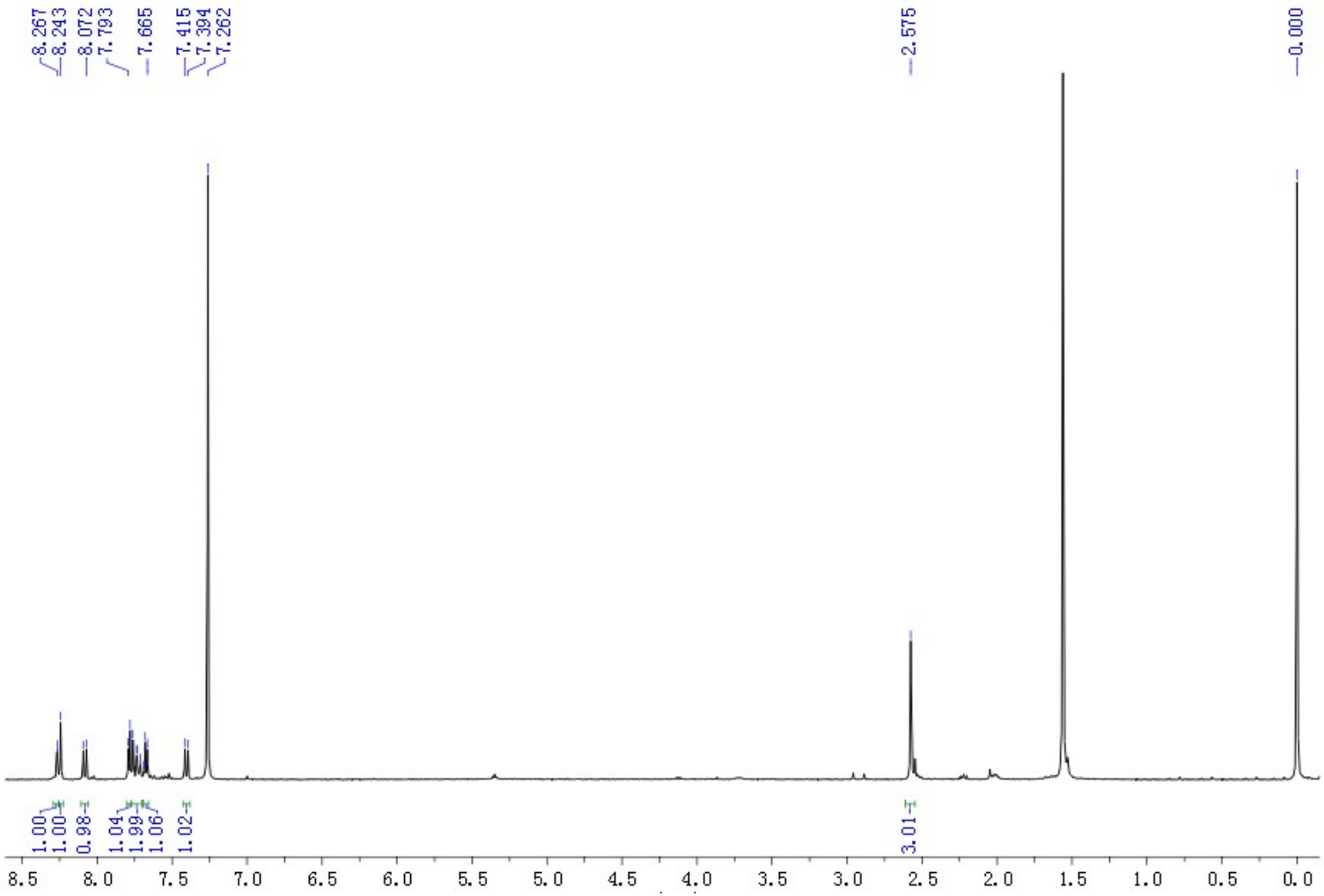
^{13}C NMR of 7-fluoro-2-methyl-16*H*-dibenzo[2,3:6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3r**)



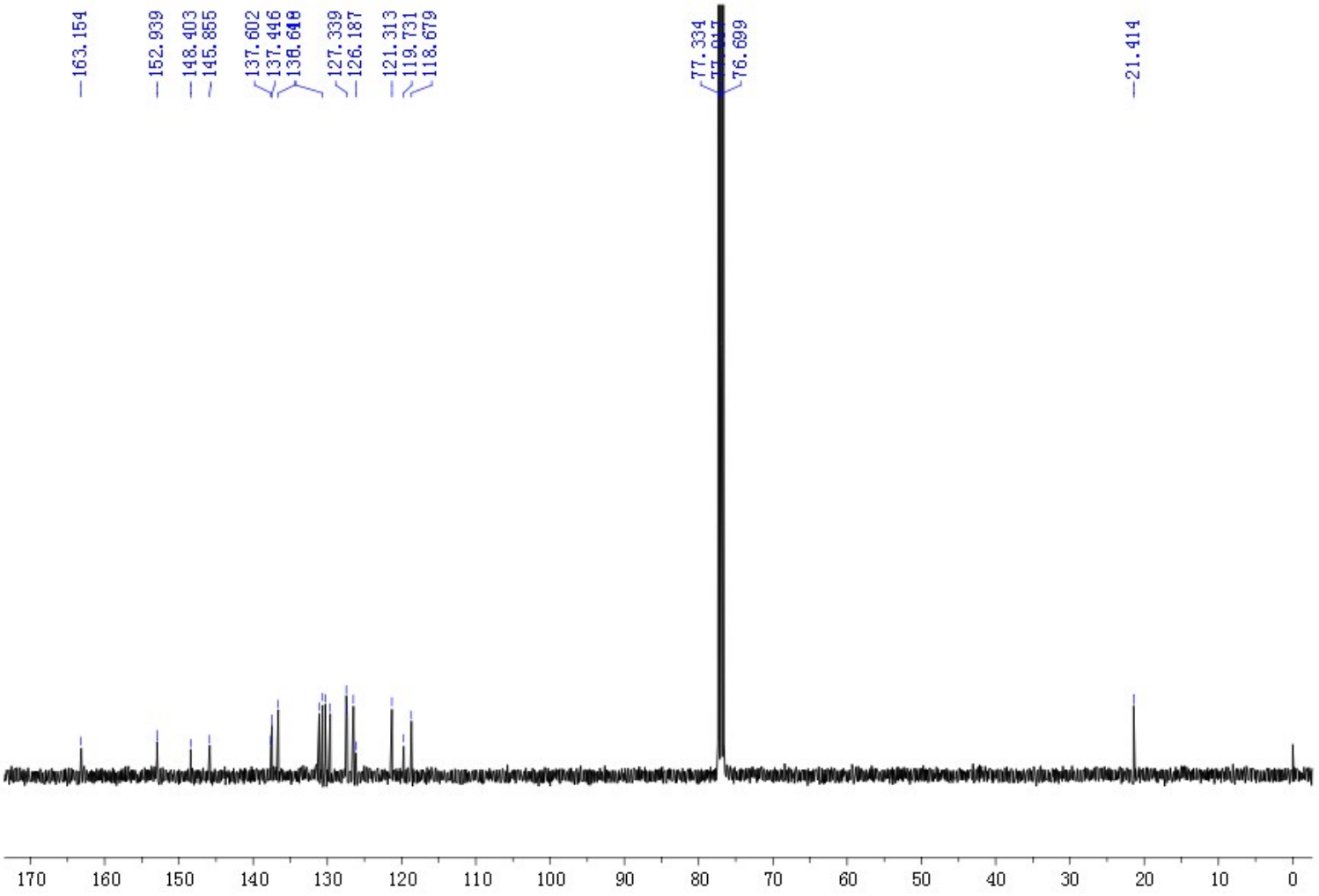
^{1}H NMR of 15*H*-benzo[2,3]thieno[2',3':6,7][1,4]oxazepino[5,4-*b*]quinazolin-15-one (**3s**)



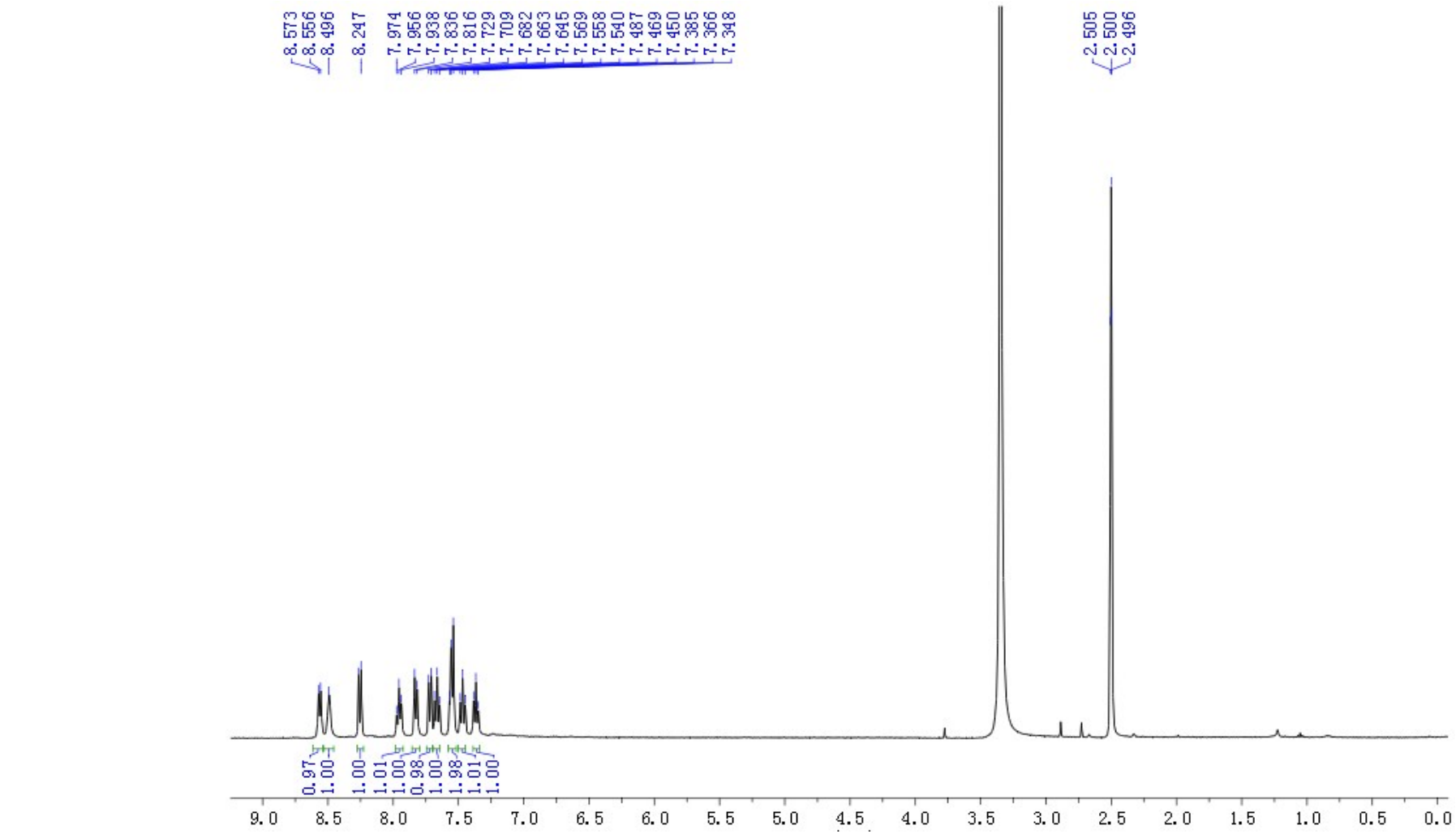
^{13}C NMR of 15*H*-benzo[2,3]thieno[2',3':6,7][1,4]oxazepino[5,4-*b*]quinazolin-15-one (**3s**)



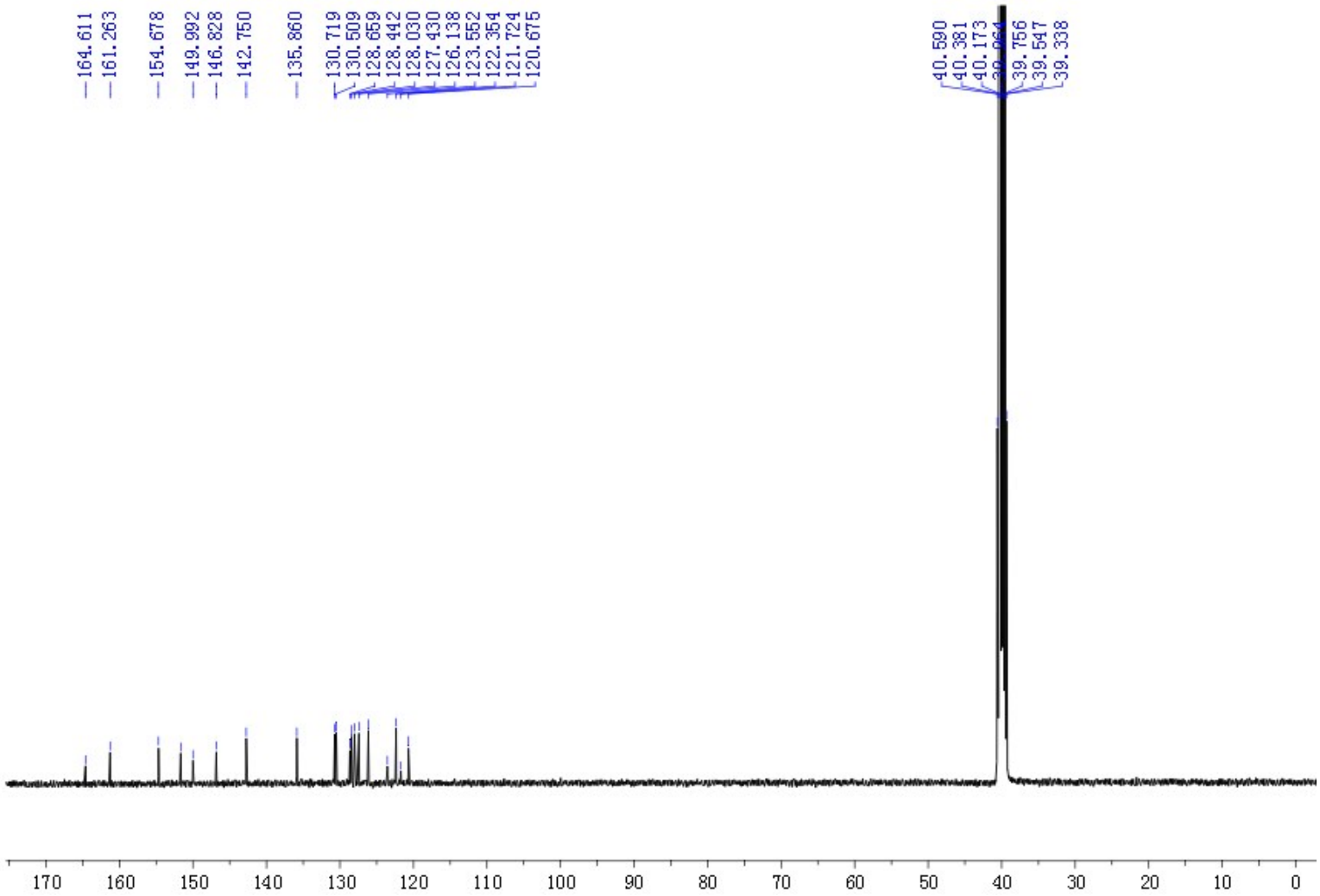
^{1}H NMR of 2-bromo-11-methyl-15*H*-benzo[2,3]thieno[2',3':6,7][1,4]oxazepino[5,4-*b*]quinazolin-15-one (**3t**)



^{13}C NMR of 2-bromo-11-methyl-15*H*-benzo[2,3]thieno[2',3':6,7][1,4]oxazepino[5,4-*b*]quinazolin-15-one (**3t**)



^{1}H NMR of 16*H*-benzo[2,3]pyrido[3',2':6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3u**)



^{13}C NMR of 16*H*-benzo[2,3]pyrido[3',2':6,7][1,4]oxazepino[5,4-*b*]quinazolin-16-one (**3u**)

CIF of the product 3c

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data_3c

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_chemical_name_systematic
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;
_chemical_name_common                   ?
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_chemical_formula_moiety                ?
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 'C20 H11 Cl N2 O2'
_chemical_formula_weight                346.76

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 _atom_type_scat_dispersion_real
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 'C'   'C'    0.0033    0.0016
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 'H'   'H'    0.0000    0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'   'N'    0.0061    0.0033
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  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'O'    'O'      0.0106      0.0060
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  'Cl'    'Cl'      0.1484      0.1585
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting                  Triclinic
_symmetry_space_group_name_H-M          P-1

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  'x, y, z'
  '-x, -y, -z'

_cell_length_a                          8.8729(11)
_cell_length_b                          9.3458(12)
_cell_length_c                          10.6991(14)
_cell_angle_alpha                       68.9710(10)
_cell_angle_beta                        88.6400(10)
_cell_angle_gamma                       69.7930(10)
_cell_volume                            772.05(17)
_cell_formula_units_Z                   2
_cell_measurement_temperature           296(2)
_cell_measurement_reflns_used           2307
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_cell_measurement_theta_max             27.99
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_exptl_crystal_density_diffrn           1.492
_exptl_crystal_density_method           'not measured'
_exptl_crystal_F_000                    356
_exptl_absorpt_coefficient_mu           0.264
_exptl_absorpt_correction_type          none
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_exptl_special_details
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_diffrn_ambient_temperature             296(2)
_diffrn_radiation_wavelength            0.71073
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_diffrn_reflns_av_sigmaI/netI       0.0251
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_computing_data_reduction           'Bruker SAINT'
_computing_structure_solution       'SHELXS-97 (Sheldrick, 1990)'
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_computing_molecular_graphics         'Bruker SHELXTL'
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 Refinement of F^2^ against ALL reflections.    The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.    R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
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_refine_ls_matrix_type                full
_refine_ls_weighting_scheme           calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0479P)^2^+0.1829P] where P=(Fo^2^+2Fc^2^)/3'
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_atom_sites_solution_secondary        difmap
_atom_sites_solution_hydrogens        geom
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Cl1 Cl -0.00165(5) 1.19380(6) 0.05888(5) 0.05628(17) Uani 1 1 d . . .
O1 O 0.82342(15) 0.28858(16) 0.34055(14) 0.0534(3) Uani 1 1 d . . .
O2 O 0.51863(14) 0.67680(15) 0.47199(11) 0.0445(3) Uani 1 1 d . . .
N1 N 0.36048(16) 0.57092(18) 0.17478(14) 0.0401(3) Uani 1 1 d . . .
N2 N 0.61148(15) 0.53334(17) 0.27849(13) 0.0352(3) Uani 1 1 d . . .
C1 C 0.44802(18) 0.6193(2) 0.23334(16) 0.0350(4) Uani 1 1 d . . .
C2 C 0.68697(19) 0.3707(2) 0.28527(16) 0.0386(4) Uani 1 1 d . . .
C3 C 0.5893(2) 0.3154(2) 0.21867(16) 0.0386(4) Uani 1 1 d . . .
C4 C 0.6538(2) 0.1605(2) 0.21080(19) 0.0476(4) Uani 1 1 d . . .
H4A H 0.7567 0.0891 0.2528 0.057 Uiso 1 1 calc R . .
C5 C 0.5639(3) 0.1140(2) 0.1403(2) 0.0536(5) Uani 1 1 d . . .
H5A H 0.6059 0.0106 0.1351 0.064 Uiso 1 1 calc R . .
C6 C 0.4112(3) 0.2210(3) 0.0771(2) 0.0533(5) Uani 1 1 d . . .
H6A H 0.3532 0.1902 0.0267 0.064 Uiso 1 1 calc R . .
C7 C 0.3439(2) 0.3719(2) 0.08771(19) 0.0489(5) Uani 1 1 d . . .
H7A H 0.2398 0.4412 0.0470 0.059 Uiso 1 1 calc R . .
C8 C 0.4329(2) 0.4209(2) 0.16028(16) 0.0387(4) Uani 1 1 d . . .
C9 C 0.36374(19) 0.7741(2) 0.25556(16) 0.0364(4) Uani 1 1 d . . .
C10 C 0.23864(19) 0.8980(2) 0.15947(17) 0.0386(4) Uani 1 1 d . . .
H10A H 0.2174 0.8879 0.0790 0.046 Uiso 1 1 calc R . .
C11 C 0.14667(19) 1.0354(2) 0.18422(18) 0.0420(4) Uani 1 1 d . . .
C12 C 0.1715(2) 1.0523(2) 0.30441(19) 0.0484(4) Uani 1 1 d . . .
H12A H 0.1053 1.1438 0.3210 0.058 Uiso 1 1 calc R . .
C13 C 0.2963(2) 0.9310(2) 0.39912(18) 0.0464(4) Uani 1 1 d . . .
H13A H 0.3154 0.9407 0.4802 0.056 Uiso 1 1 calc R . .
C14 C 0.3926(2) 0.7954(2) 0.37394(16) 0.0391(4) Uani 1 1 d . . .

C15 C 0.6676(2) 0.6666(2) 0.42273(17) 0.0416(4) Uani 1 1 d . . .
C16 C 0.7677(3) 0.7239(2) 0.4719(2) 0.0554(5) Uani 1 1 d . . .
H16A H 0.7384 0.7641 0.5403 0.066 Uiso 1 1 calc R . .
C17 C 0.9110(3) 0.7213(3) 0.4190(2) 0.0640(6) Uani 1 1 d . . .
H17A H 0.9799 0.7577 0.4530 0.077 Uiso 1 1 calc R . .
C18 C 0.9528(2) 0.6649(3) 0.3160(2) 0.0596(6) Uani 1 1 d . . .
H18A H 1.0480 0.6670 0.2784 0.071 Uiso 1 1 calc R . .
C19 C 0.8537(2) 0.6051(2) 0.26800(19) 0.0480(4) Uani 1 1 d . . .
H19A H 0.8830 0.5656 0.1992 0.058 Uiso 1 1 calc R . .
C20 C 0.71050(19) 0.6043(2) 0.32281(16) 0.0383(4) Uani 1 1 d . . .

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O1 0.0429(7) 0.0504(8) 0.0629(8) -0.0268(7) -0.0053(6) -0.0055(6)
O2 0.0542(7) 0.0414(7) 0.0329(6) -0.0129(5) 0.0004(5) -0.0119(6)
N1 0.0387(7) 0.0420(8) 0.0440(8) -0.0190(7) 0.0029(6) -0.0164(6)
N2 0.0352(7) 0.0374(8) 0.0359(7) -0.0155(6) 0.0038(5) -0.0146(6)
C1 0.0360(8) 0.0373(9) 0.0328(8) -0.0128(7) 0.0036(6) -0.0150(7)
C2 0.0393(9) 0.0397(10) 0.0368(9) -0.0159(8) 0.0069(7) -0.0128(7)

C3 0.0453(9) 0.0392(9) 0.0356(9) -0.0155(8) 0.0104(7) -0.0192(8)
C4 0.0563(11) 0.0420(10) 0.0482(10) -0.0200(9) 0.0132(8) -0.0192(9)
C5 0.0742(13) 0.0443(11) 0.0579(12) -0.0279(10) 0.0234(10) -0.0316(10)
C6 0.0693(13) 0.0584(13) 0.0534(11) -0.0292(10) 0.0138(10) -0.0398(11)
C7 0.0521(10) 0.0531(12) 0.0523(11) -0.0236(9) 0.0063(8) -0.0275(9)
C8 0.0451(9) 0.0412(10) 0.0366(9) -0.0168(8) 0.0089(7) -0.0215(8)
C9 0.0363(8) 0.0389(9) 0.0382(9) -0.0158(7) 0.0066(7) -0.0172(7)
C10 0.0373(8) 0.0433(10) 0.0390(9) -0.0167(8) 0.0038(7) -0.0175(7)
C11 0.0347(8) 0.0413(10) 0.0466(10) -0.0132(8) 0.0048(7) -0.0132(7)
C12 0.0473(10) 0.0444(11) 0.0534(11) -0.0242(9) 0.0094(8) -0.0103(8)
C13 0.0555(10) 0.0463(11) 0.0397(9) -0.0215(8) 0.0076(8) -0.0157(9)
C14 0.0440(9) 0.0373(9) 0.0351(9) -0.0122(7) 0.0048(7) -0.0151(7)
C15 0.0458(9) 0.0339(9) 0.0393(9) -0.0092(8) -0.0073(7) -0.0116(7)
C16 0.0666(13) 0.0412(11) 0.0545(12) -0.0165(9) -0.0194(10) -0.0146(9)
C17 0.0636(13) 0.0498(12) 0.0752(15) -0.0116(11) -0.0260(11) -0.0264(10)
C18 0.0445(10) 0.0569(13) 0.0683(13) -0.0055(11) -0.0082(9) -0.0254(9)
C19 0.0435(9) 0.0493(11) 0.0474(10) -0.0104(9) -0.0001(8) -0.0198(8)
C20 0.0393(8) 0.0369(9) 0.0376(9) -0.0102(7) -0.0038(7) -0.0157(7)

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All esds (except the esd in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell esds are taken
into account individually in the estimation of esds in distances, angles
and torsion angles; correlations between esds in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)

```
  treatment of cell esds is used for estimating esds involving l.s. planes.
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O1 C2 1.213(2) . ?
O2 C14 1.395(2) . ?
O2 C15 1.396(2) . ?
N1 C1 1.292(2) . ?
N1 C8 1.390(2) . ?
N2 C1 1.390(2) . ?
N2 C2 1.408(2) . ?
N2 C20 1.444(2) . ?
C1 C9 1.483(2) . ?
C2 C3 1.459(2) . ?
C3 C4 1.394(2) . ?
C3 C8 1.394(2) . ?
C4 C5 1.377(3) . ?
C4 H4A 0.9300 . ?
C5 C6 1.384(3) . ?
C5 H5A 0.9300 . ?
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C6 C7 1.374(3) . ?
C6 H6A 0.9300 . ?
C7 C8 1.404(2) . ?
C7 H7A 0.9300 . ?
C9 C14 1.393(2) . ?
C9 C10 1.393(2) . ?
C10 C11 1.376(2) . ?
C10 H10A 0.9300 . ?
C11 C12 1.382(3) . ?
C12 C13 1.378(3) . ?
C12 H12A 0.9300 . ?
C13 C14 1.376(2) . ?
C13 H13A 0.9300 . ?
C15 C20 1.379(2) . ?
C15 C16 1.381(2) . ?
C16 C17 1.376(3) . ?
C16 H16A 0.9300 . ?
C17 C18 1.375(3) . ?
C17 H17A 0.9300 . ?
C18 C19 1.384(3) . ?
C18 H18A 0.9300 . ?
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C1 N1 C8 117.94(14) . . ?
C1 N2 C2 120.92(13) . . ?
C1 N2 C20 122.01(13) . . ?
C2 N2 C20 117.06(13) . . ?
N1 C1 N2 124.04(15) . . ?
N1 C1 C9 116.35(14) . . ?
N2 C1 C9 119.58(14) . . ?
O1 C2 N2 120.86(15) . . ?
O1 C2 C3 124.66(16) . . ?
N2 C2 C3 114.47(14) . . ?
C4 C3 C8 120.71(16) . . ?
C4 C3 C2 120.13(16) . . ?
C8 C3 C2 119.14(15) . . ?
C5 C4 C3 119.49(18) . . ?
C5 C4 H4A 120.3 . . ?
C3 C4 H4A 120.3 . . ?
C4 C5 C6 120.08(18) . . ?
C4 C5 H5A 120.0 . . ?
C6 C5 H5A 120.0 . . ?
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C10 C11 Cl1 118.92(13) . . ?
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C17 C16 C15 119.6(2) . . ?
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C18 C19 H19A 120.1 . . ?
C20 C19 H19A 120.1 . . ?
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C15 C20 N2 120.79(15) . . ?
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C2 N2 C1 N1 -11.7(2) . . . . ?
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C2 N2 C1 C9 166.40(14) . . . . ?
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O1 C2 C3 C8 179.12(16) . . . . ?
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C1 N1 C8 C7 -175.07(15) . . . . ?
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C4 C3 C8 N1 175.09(15) . . . . ?
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C6 C7 C8 C3 0.8(3) . . . . ?
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N1 C1 C9 C10 -34.6(2) . . . . ?
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C16 C15 C20 C19 -2.3(3) . . . . ?
O2 C15 C20 C19 175.57(15) . . . . ?
C16 C15 C20 N2 175.42(16) . . . . ?
O2 C15 C20 N2 -6.7(2) . . . . ?
C18 C19 C20 C15 1.4(3) . . . . ?
C18 C19 C20 N2 -176.42(16) . . . . ?
C1 N2 C20 C15 55.0(2) . . . . ?
C2 N2 C20 C15 -123.82(17) . . . . ?
C1 N2 C20 C19 -127.28(17) . . . . ?
C2 N2 C20 C19 53.9(2) . . . . ?

loop_
  _geom_hbond_atom_site_label_D
  _geom_hbond_atom_site_label_H
  _geom_hbond_atom_site_label_A
  _geom_hbond_distance_DH
  _geom_hbond_distance_HA
  _geom_hbond_distance_DA
  _geom_hbond_angle_DHA
  _geom_hbond_site_symmetry_A
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C10 H10A Cl1 0.93 2.89 3.6787(17) 142.9 2_575
C12 H12A O1 0.93 2.44 3.226(2) 141.8 1_465

_diffrn_measured_fraction_theta_max 0.984
_diffrn_reflns_theta_full 25.01
_diffrn_measured_fraction_theta_full 0.984
_refine_diff_density_max 0.157
_refine_diff_density_min -0.205
_refine_diff_density_rms 0.036