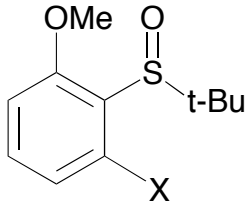
Supplementary Material for Organic & Biomolecular Chemistry

Supplementary Information for B716954J

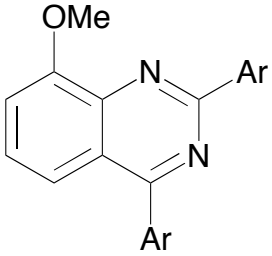
^{13}C Spectra for compounds in Scheme 1, 2, 3

CIF files for 4b, 4d, 4f

13-C spectra deposited in the Supplementary Information

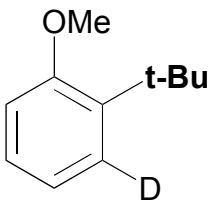


3, X = D
4a, X = $CONMe_2$; **4d**, X = CH_2OH;
4b, X = CHO; **4e**, X = $B(OCMe_2CMe_2O)$;
4c, X = Br; **4f**, X = Me.



5 Ar = Ph, 85%

7 Ar = 3,5-$CF_3C_6H_3$, 45%



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Chemist JPF
Group JMB
Expt 1.29
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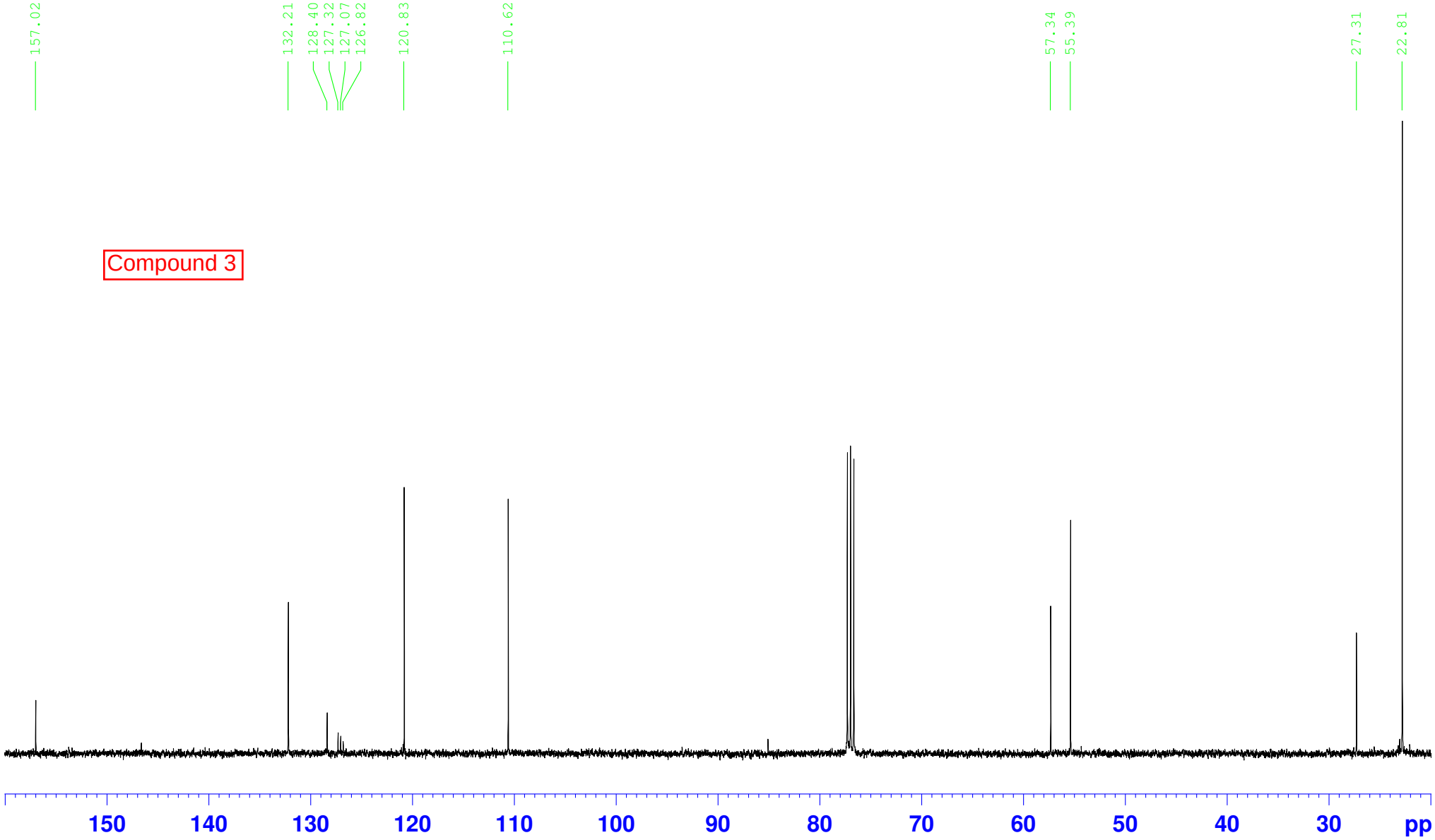

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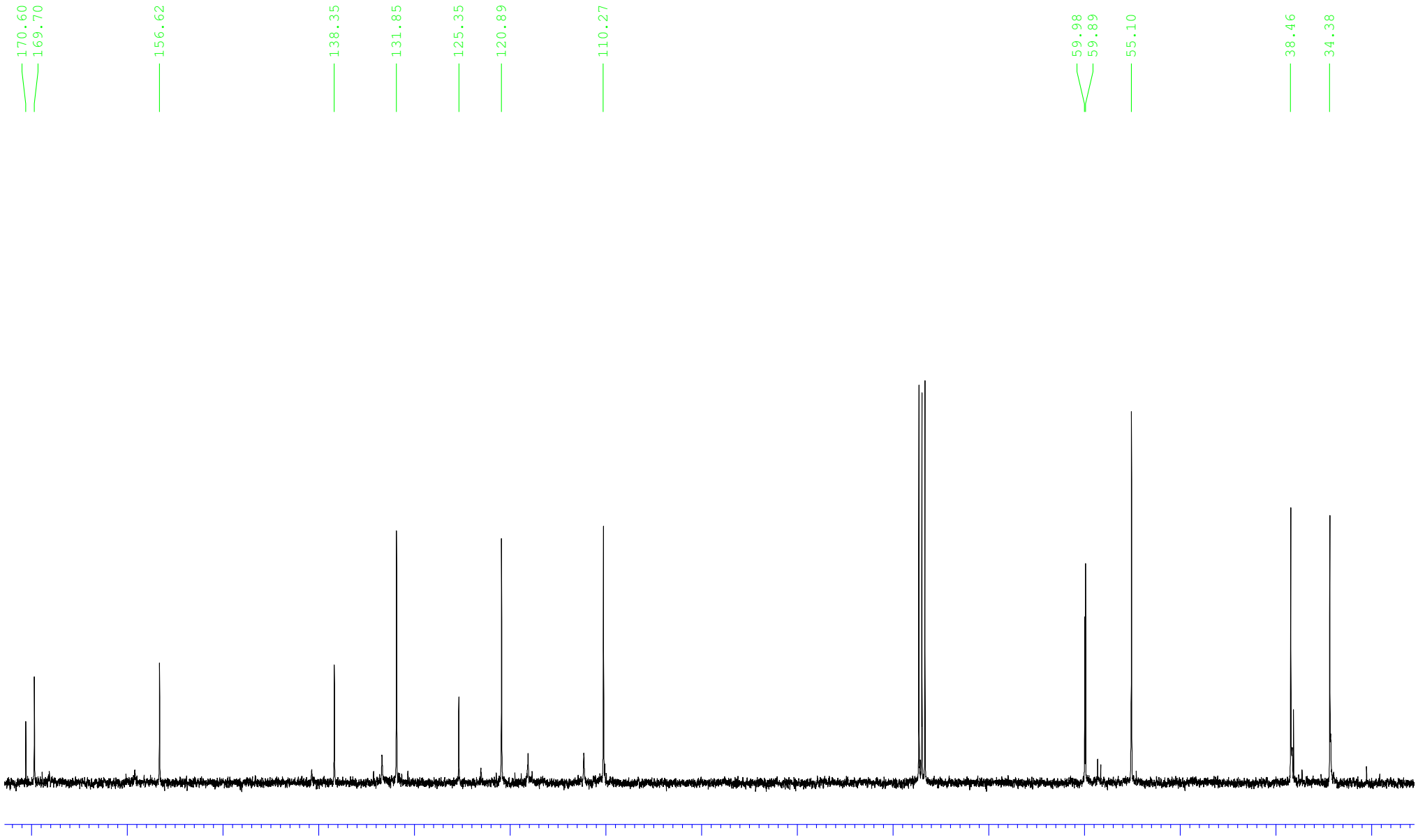

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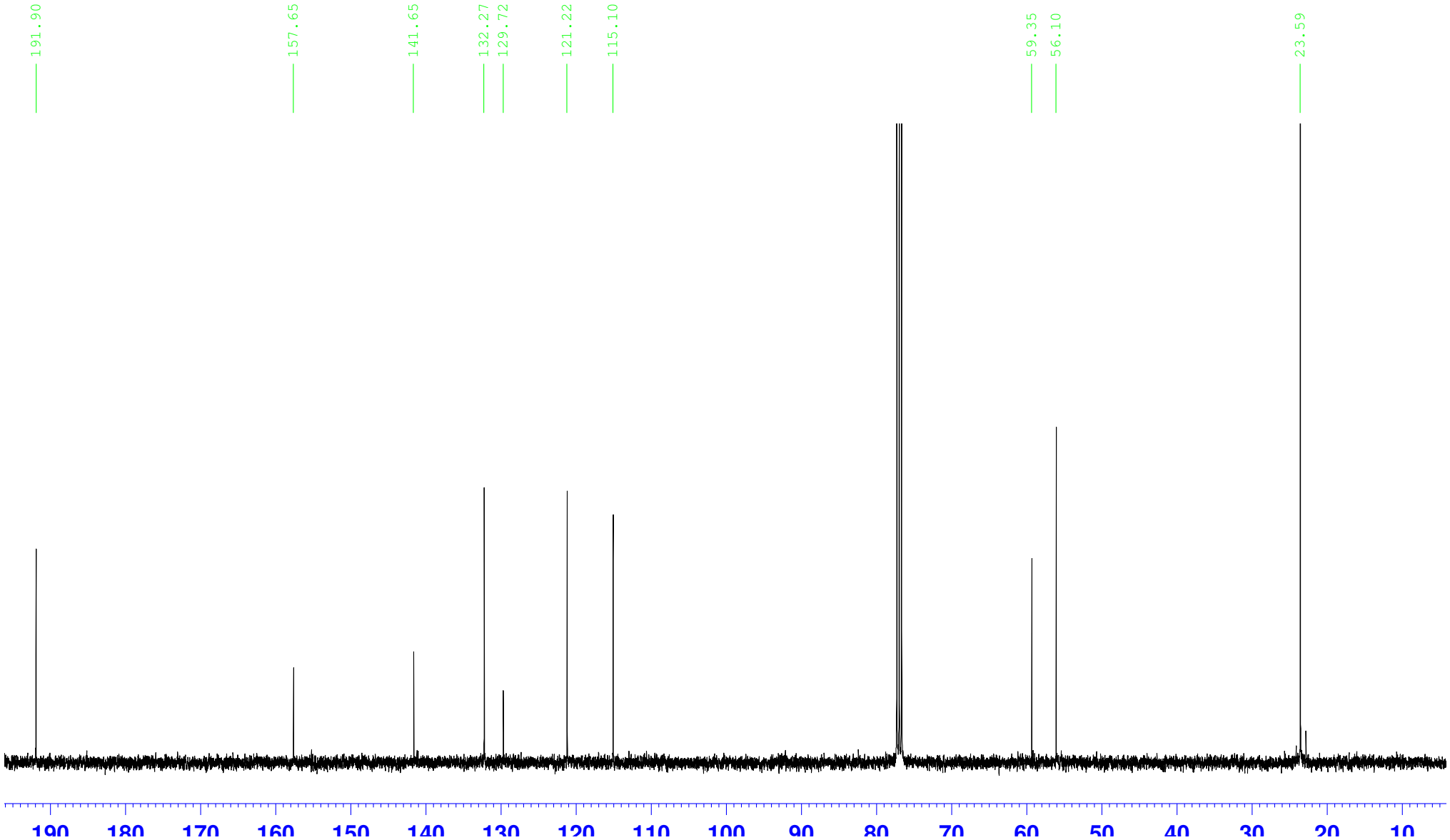

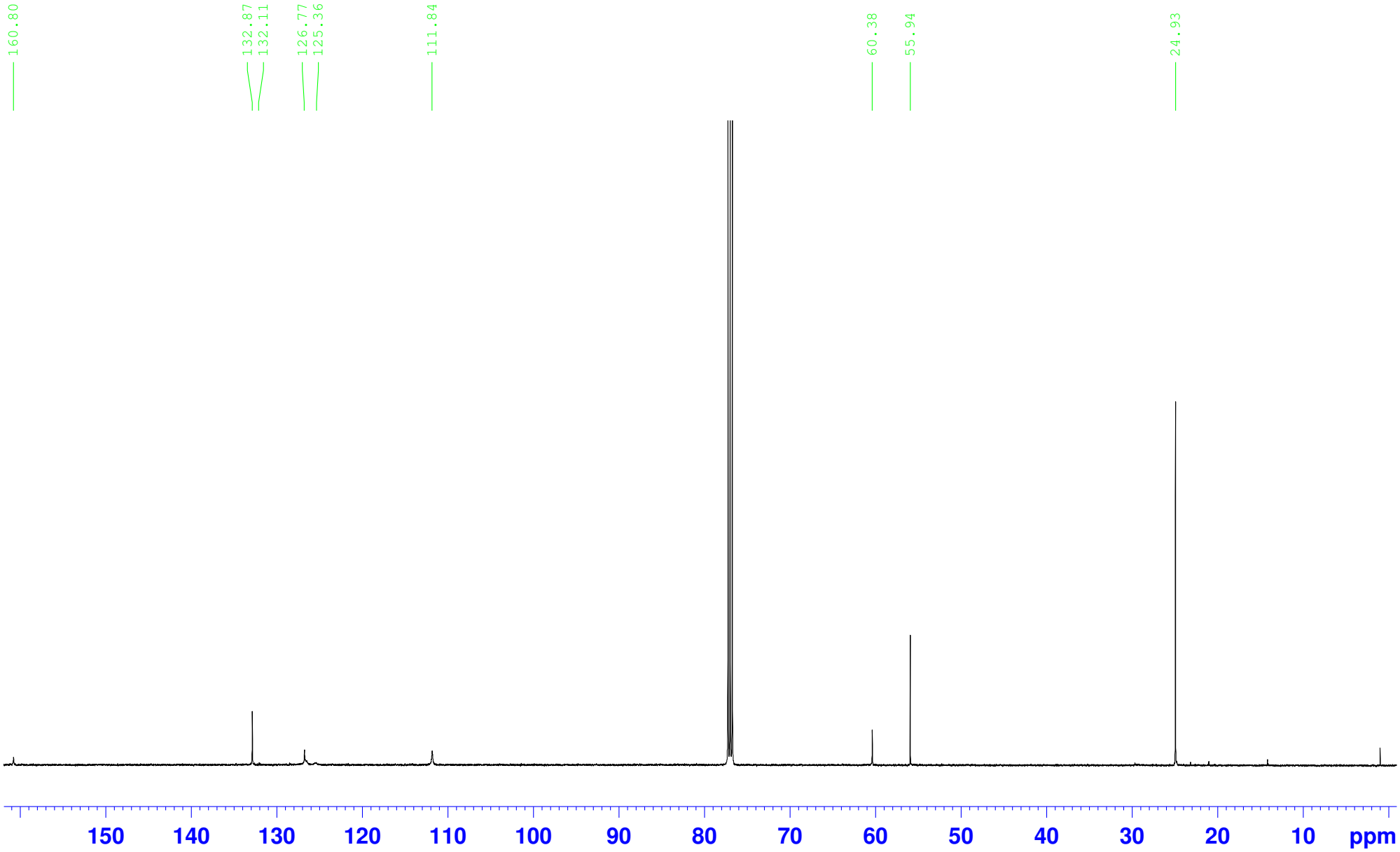
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90
80
70
60
50
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20
10
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Instrument DQX400
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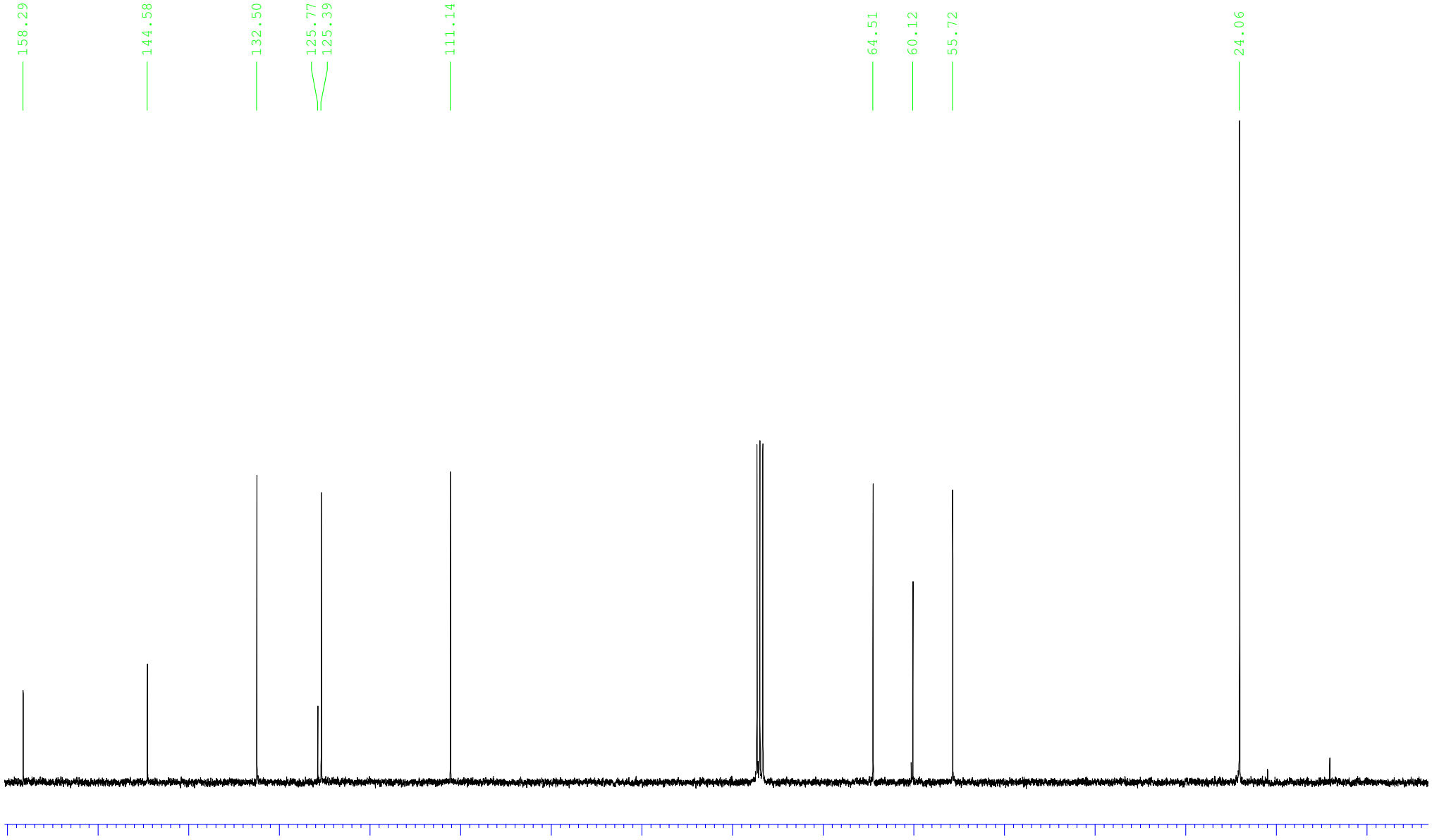
Group JMB
Expt 2.26 after column
c13acq.au CDCl3 e:\\ jmbgrp 56



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Group JMB
Expt 8.43
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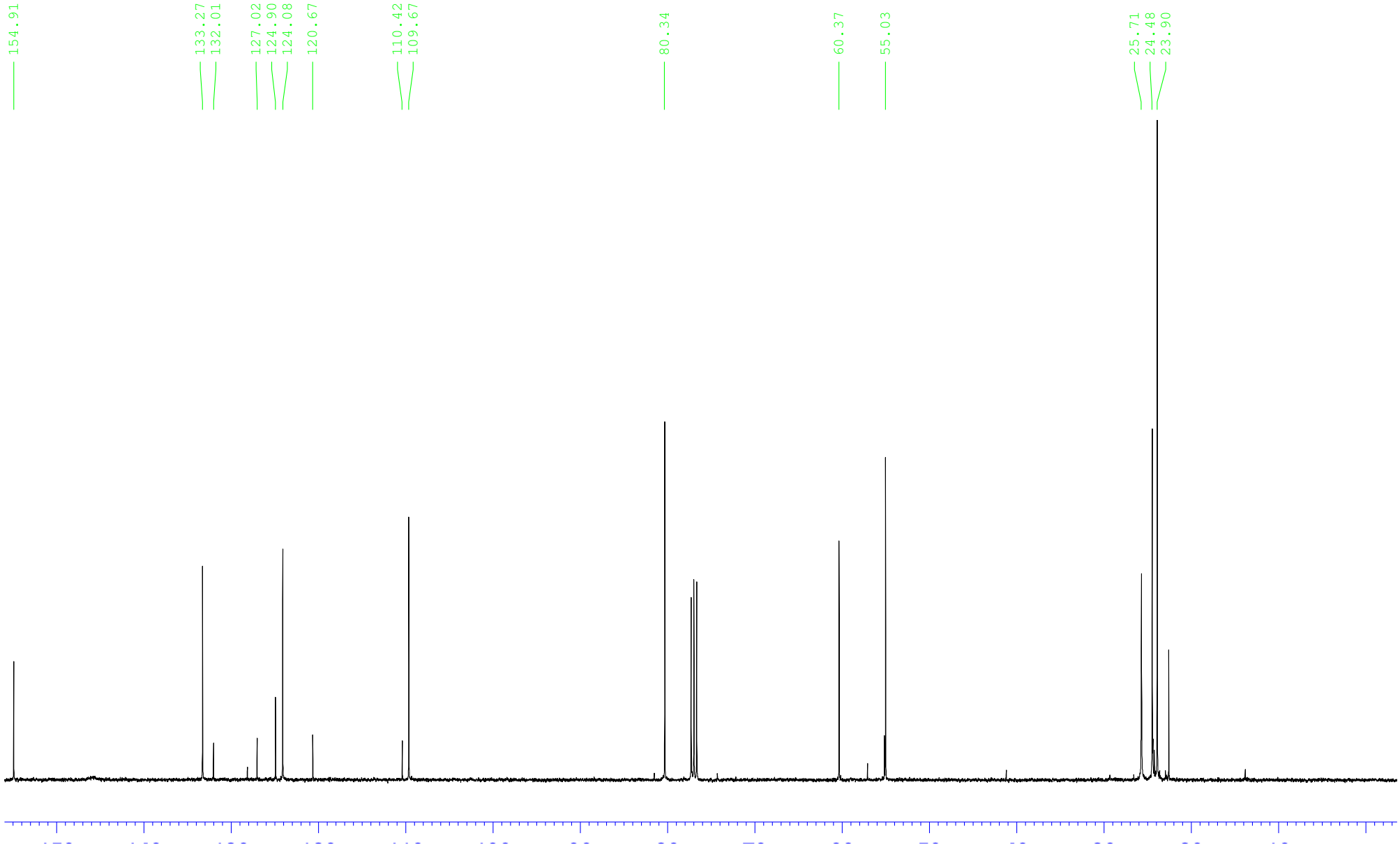

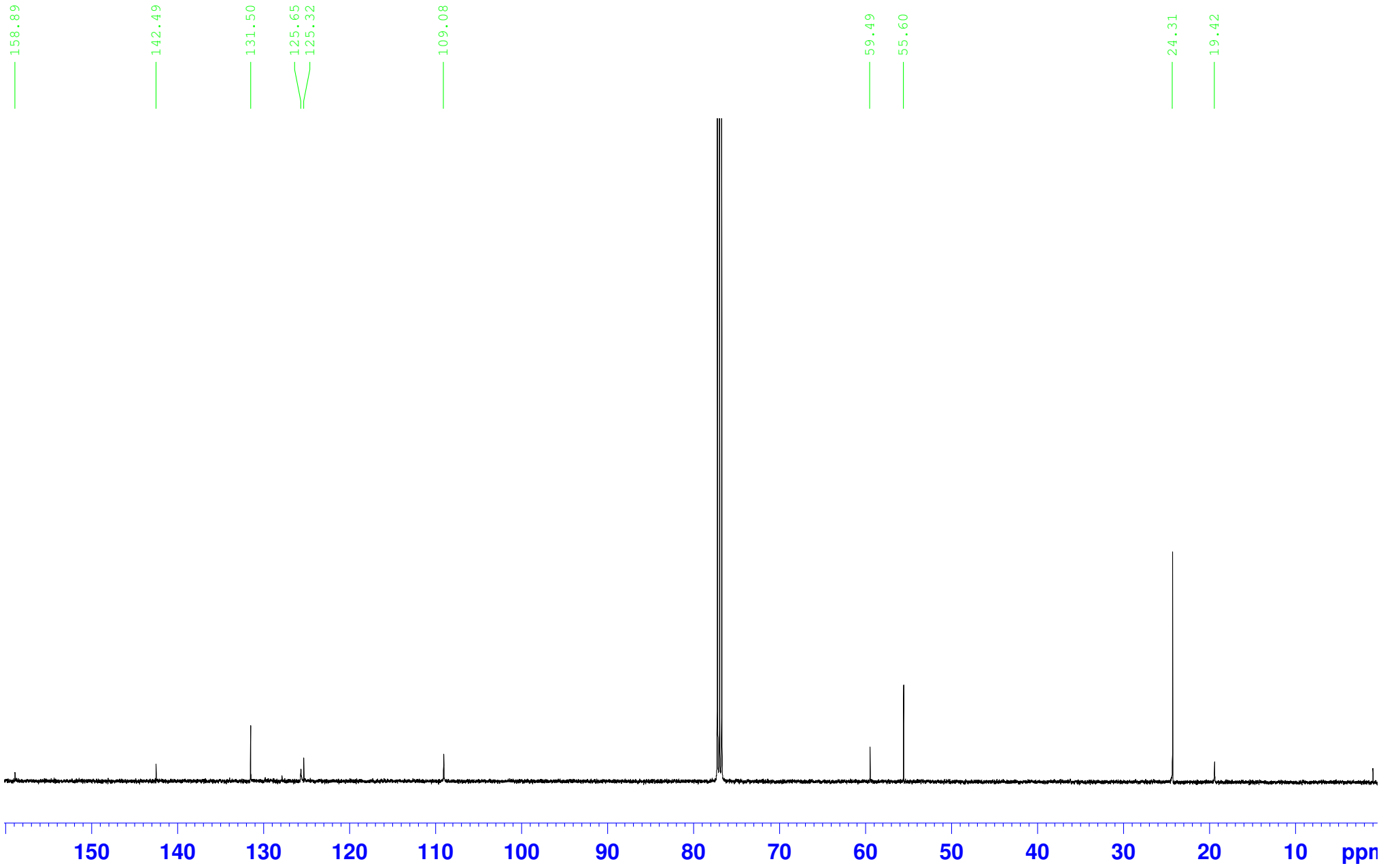
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100
90
80
70
60
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ppm

Instrument DQX400
Chemist JPF
Group JMB
Expt 2.58
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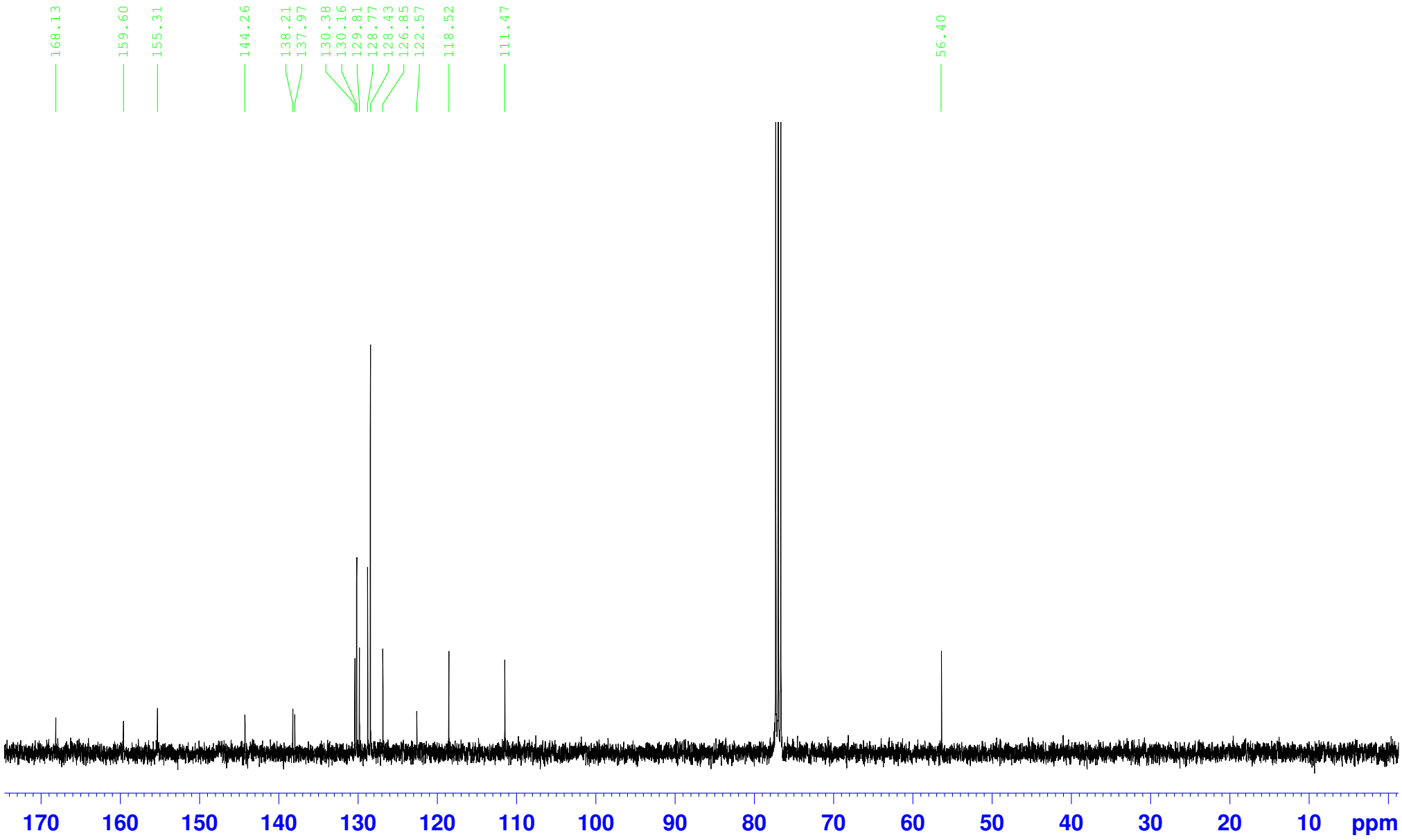

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Expt 9.01 pCF3 crystals
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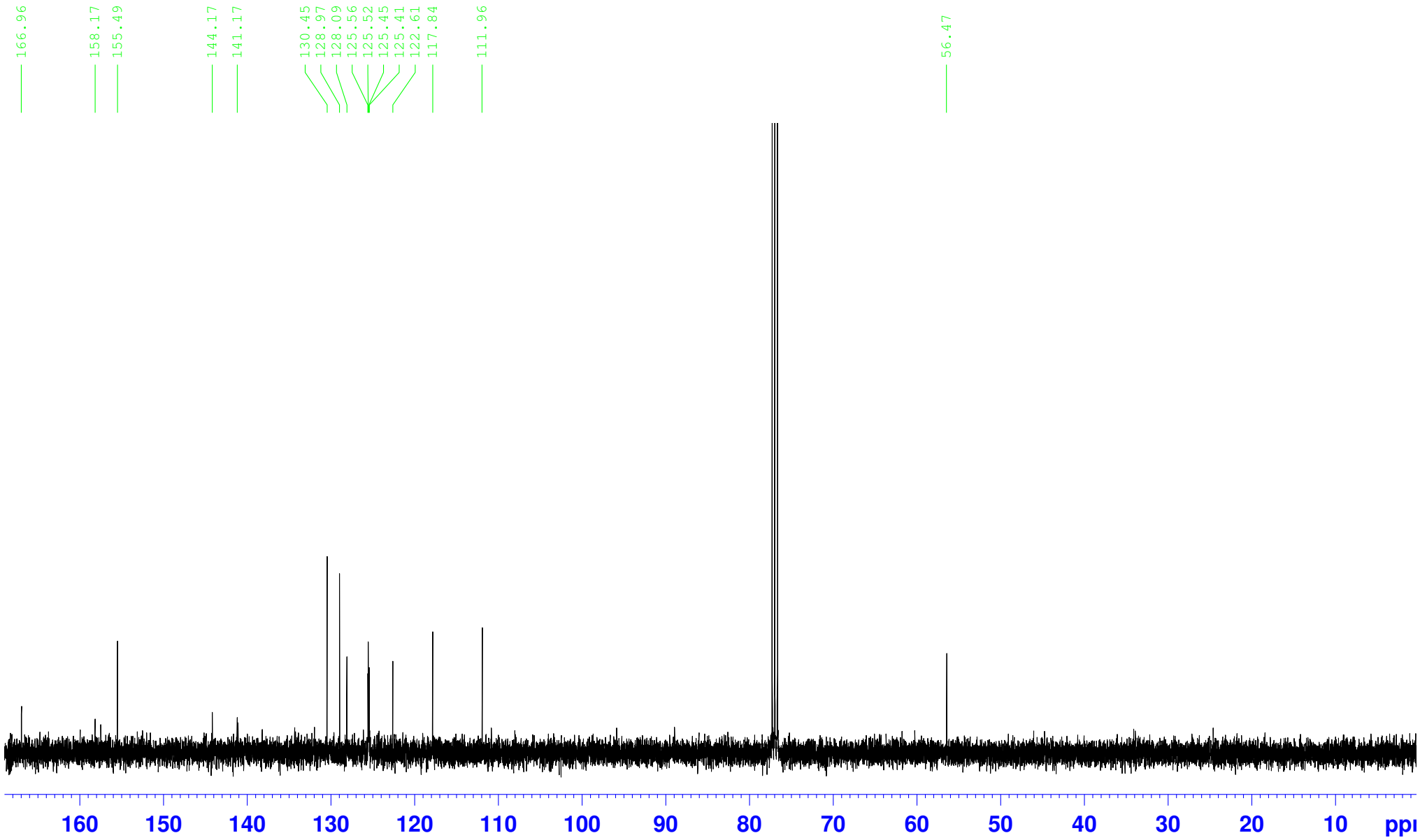

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Expt 1.32 spot 1
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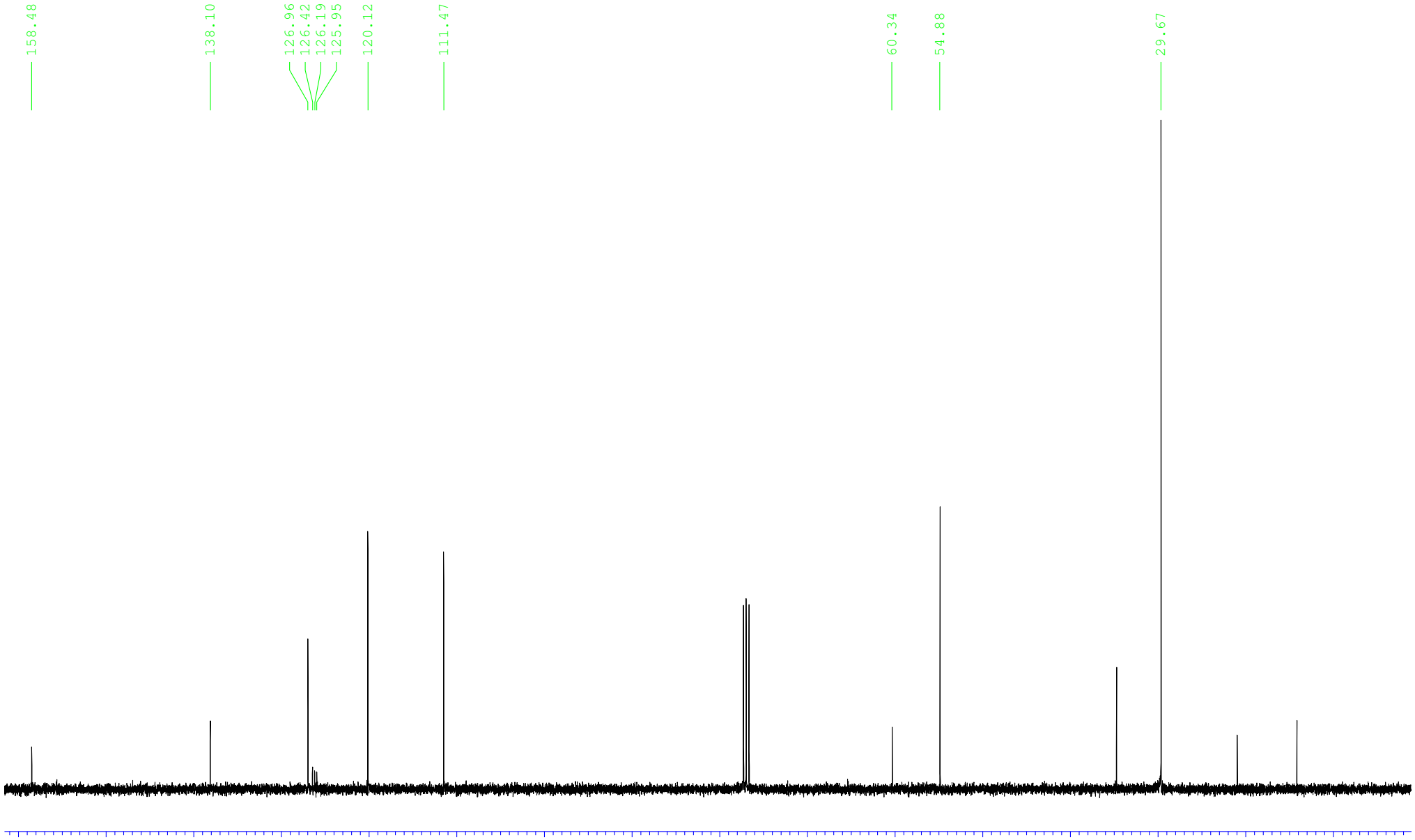

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Oxford  University
Mansfield Rd.
Oxford OX1 3TA
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Sequential ortho-lithiations; the sulfoxide group
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Gunnels Wood Rd.
Stevenage
Herts SG1 2NY
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Sulfoxides are known to be powerful directing groups for ortho-lithiation,
even in competition with other directors.This has been utilised to
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substituents meta- to a methoxy-group by sequential lithiation,
reaction with Me tert-butylsulfinate, and a second lithiation.
```

```
Electrophilic trapping of the ensuing lithio-compound
with a range of electrophiles followed by reductive removal of the
sulfoxide
led to meta-substituted anisoles.
The X-ray structure of the hydroxymethyl compound is included here.
Some interesting side-reactions were uncovered,including a short synthesis
of quinazolines arising from the use of PhCN in the second step.
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 Method, part 1, Chebychev polynomial, (Watkin, 1994, Prince, 1982)
 [weight] = 1.0/[A~0~*T~0~(x)+A~1~*T~1~(x) ... +A~n-1~]*T~n-1~(x)]
 where A~i~ are the Chebychev coefficients listed below and x= Fcalc/Fmax
 Method = Robust Weighting (Prince, 1982)
 W = [weight] * [1-(deltaF/6*sigmaF)^2^]^2^
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 1.81 0.498 1.49
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C2 C 0.5605(3) 0.4875(2) 0.33862(19) 0.0321 1.0000 Uani . . . . . .
C3 C 0.4523(3) 0.5572(2) 0.1485(2) 0.0393 1.0000 Uani . . . . . .
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C11 C 0.0201(3) 0.4047(2) 0.3927(2) 0.0421 1.0000 Uani . . . . . .
O2 O -0.1078(3) 0.3779(2) 0.4134(3) 0.0836 1.0000 Uani . . . . . .
O3 O 0.51726(18) 0.22846(14) 0.35285(15) 0.0363 1.0000 Uani . . . . . .
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H22 H 0.5349 0.4484 0.4124 0.0383 1.0000 Uiso . . . . . .
H23 H 0.6281 0.4381 0.2923 0.0383 1.0000 Uiso . . . . . .
H31 H 0.5065 0.6310 0.1568 0.0471 1.0000 Uiso . . . . . .
H32 H 0.5186 0.5020 0.1082 0.0471 1.0000 Uiso . . . . . .
H33 H 0.3560 0.5683 0.1018 0.0471 1.0000 Uiso . . . . . .
H41 H 0.3661 0.6641 0.3494 0.0403 1.0000 Uiso . . . . . .
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H81 H 0.2419 0.1875 0.6720 0.0454 1.0000 Uiso . . . . . .
H91 H 0.0411 0.2982 0.5901 0.0443 1.0000 Uiso . . . . . .
H111 H 0.0383 0.4731 0.3442 0.0504 1.0000 Uiso . . . . . .
H121 H 0.7055 0.1371 0.3297 0.0615 1.0000 Uiso . . . . . .
H122 H 0.6705 0.1630 0.4656 0.0615 1.0000 Uiso . . . . . .
H123 H 0.5705 0.0687 0.3939 0.0615 1.0000 Uiso . . . . . .
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C9 0.0368(13) 0.0365(12) 0.0375(13) -0.0037(10) 0.0051(10) -0.0090(10)
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C4 . H43 . 1.000    no
C5 . C6 . 1.403(3)    yes
C5 . C10 . 1.398(3)    yes
C6 . C7 . 1.390(3)    yes
C6 . O3 . 1.367(3)    yes
C7 . C8 . 1.381(4)    yes
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C8 . C9 . 1.386(4)    yes
C8 . H81 . 1.000    no
C9 . C10 . 1.398(3)    yes
C9 . H91 . 1.000    no
C10 . C11 . 1.485(3)    yes
C11 . O2 . 1.201(4)    yes
C11 . H111 . 1.000    no
O3 . C12 . 1.435(3)    yes
C12 . H121 . 1.000    no
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S1 . C1 . C2 . 109.31(15)    yes
S1 . C1 . C3 . 102.82(15)    yes
C2 . C1 . C3 . 110.4(2)    yes
S1 . C1 . C4 . 110.75(16)    yes
C2 . C1 . C4 . 111.99(18)    yes
C3 . C1 . C4 . 111.23(19)    yes
C1 . C2 . H21 . 109.466    no
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H21 . C2 . H22 . 109.476    no
C1 . C2 . H23 . 109.467    no
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C1 . C3 . H31 . 109.467    no
C1 . C3 . H32 . 109.467    no
H31 . C3 . H32 . 109.475    no
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H41 . C4 . H42 . 109.476    no
C1 . C4 . H43 . 109.467    no
H41 . C4 . H43 . 109.476    no
H42 . C4 . H43 . 109.476    no
S1 . C5 . C6 . 116.34(17)    yes
S1 . C5 . C10 . 123.53(17)    yes
C6 . C5 . C10 . 119.7(2)    yes
C5 . C6 . C7 . 120.4(2)    yes
C5 . C6 . O3 . 115.7(2)    yes
C7 . C6 . O3 . 123.9(2)    yes
C6 . C7 . C8 . 119.3(2)    yes
C6 . C7 . H71 . 120.350    no
C8 . C7 . H71 . 120.350    no
C7 . C8 . C9 . 120.9(2)    yes
C7 . C8 . H81 . 119.532    no
C9 . C8 . H81 . 119.532    no
C8 . C9 . C10 . 120.3(2)    yes
C8 . C9 . H91 . 119.844    no
C10 . C9 . H91 . 119.844    no
C5 . C10 . C9 . 119.1(2)    yes
C5 . C10 . C11 . 124.2(2)    yes
C9 . C10 . C11 . 116.7(2)    yes
C10 . C11 . O2 . 121.2(3)    yes
C10 . C11 . H111 . 119.408    no
O2 . C11 . H111 . 119.408    no
C6 . O3 . C12 . 117.7(2)    yes
O3 . C12 . H121 . 109.467    no
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_publ_contact_author_address
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Chemistry Research Laboratory
Oxford  University
Mansfield Rd.
Oxford OX1 3TA
;
_publ_contact_author_phone '44-1865-275642'
_publ_contact_author_fax '44-1865-285002'
_publ_contact_author_email john.brown@chem.ox.ac.uk

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;
Sequential ortho-lithiations; the sulfoxide group
as a relay to enable meta-substitution
;
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;
Chemistry Research Laboratory
Oxford  University
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;
GlaxoSmithKline
Gunnels Wood Rd.
Stevenage
Herts SG1 2NY
;


_publ_section_abstract
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Sulfoxides are known to be powerful directing groups for ortho-lithiation,
even in competition with other directors.This has been utilised to
introduce
substituents meta- to a methoxy-group by sequential lithiation,
reaction with Me tert-butylsulfinate, and a second lithiation.
```

```
Electrophilic trapping of the ensuing lithio-compound
with a range of electrophiles followed by reductive removal of the
sulfoxide
led to meta-substituted anisoles.
The X-ray structure of the hydroxymethyl compound is included here.
Some interesting side-reactions were uncovered,including a short synthesis
of quinazolines arising from the use of PhCN in the second step.
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synthesis as described in manuscriptCCDC
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 Denzo/Scalepack (Otwinowski & Minor, 1996)
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# well as the _gt R-factors:
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_refine_ls_weighting_scheme       calc
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```
;
 Method, part 1, Chebychev polynomial, (Watkin, 1994, Prince, 1982)
 [weight] = 1.0/[A~0~*T~0~(x)+A~1~*T~1~(x) ... +A~n-1~]*T~n-1~(x)]
 where A~i~ are the Chebychev coefficients listed below and x= Fcalc/Fmax
 Method = Robust Weighting (Prince, 1982)
 W = [weight] * [1-(deltaF/6*sigmaF)^2^]^2^
 A~i~ are:
 2.85 -0.371 2.02
;
_diffrn_radiation_type            'Mo K\a'
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# Uequiv = arithmetic mean of Ui
# i.e. Ueqiv = (U1+U2+U3)/3

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C1 C 0.08243(15) 0.48957(10) 0.73136(10) 0.0181 1.0000 Uani
C2 C 0.03745(17) 0.44441(12) 0.84676(12) 0.0255 1.0000 Uani
C3 C 0.19298(16) 0.40972(11) 0.67146(12) 0.0229 1.0000 Uani
C4 C -0.06325(16) 0.51398(11) 0.66187(11) 0.0225 1.0000 Uani
C5 C 0.23041(15) 0.67657(10) 0.62896(10) 0.0168 1.0000 Uani
C6 C 0.11750(15) 0.75020(11) 0.58533(11) 0.0190 1.0000 Uani
C7 C 0.13799(16) 0.79858(12) 0.48062(11) 0.0240 1.0000 Uani
C8 C 0.27396(17) 0.77511(12) 0.42141(11) 0.0250 1.0000 Uani
C9 C 0.38954(16) 0.70735(12) 0.46634(11) 0.0223 1.0000 Uani
C10 C 0.37098(14) 0.65750(10) 0.57060(10) 0.0175 1.0000 Uani
C11 C 0.50846(16) 0.59290(12) 0.61695(11) 0.0232 1.0000 Uani
O2 O 0.58484(12) 0.65282(10) 0.70496(9) 0.0322 1.0000 Uani
O3 O -0.00983(11) 0.76943(9) 0.65152(9) 0.0267 1.0000 Uani
C12 C -0.1194(2) 0.85341(16) 0.61755(16) 0.0426 1.0000 Uani
H1 H 0.502(3) 0.640(2) 0.769(2) 0.075(8) 1.0000 Uiso
H21 H -0.0187 0.3716 0.8381 0.0306 1.0000 Uiso
H22 H -0.0327 0.4992 0.8851 0.0306 1.0000 Uiso
H23 H 0.1345 0.4330 0.8922 0.0306 1.0000 Uiso
H31 H 0.1362 0.3388 0.6539 0.0275 1.0000 Uiso
H32 H 0.2850 0.3930 0.7205 0.0275 1.0000 Uiso
H33 H 0.2304 0.4449 0.6007 0.0275 1.0000 Uiso
H41 H -0.1177 0.4423 0.6435 0.0270 1.0000 Uiso
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H42 H -0.1359 0.5632 0.7052 0.0270 1.0000 Uiso
H43 H -0.0318 0.5525 0.5913 0.0270 1.0000 Uiso
H71 H 0.0562 0.8493 0.4488 0.0288 1.0000 Uiso
H81 H 0.2887 0.8075 0.3453 0.0300 1.0000 Uiso
H91 H 0.4882 0.6939 0.4232 0.0268 1.0000 Uiso
H111 H 0.5857 0.5791 0.5557 0.0278 1.0000 Uiso
H112 H 0.4699 0.5199 0.6467 0.0278 1.0000 Uiso
H121 H -0.2062 0.8584 0.6735 0.0512 1.0000 Uiso
H122 H -0.0647 0.9271 0.6124 0.0512 1.0000 Uiso
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O1 0.0224(5) 0.0310(5) 0.0203(5) 0.0027(4) -0.0087(4) -0.0016(4)
C1 0.0180(6) 0.0163(5) 0.0200(6) 0.0008(5) 0.0014(4) -0.0005(5)
C2 0.0264(7) 0.0246(7) 0.0255(7) 0.0069(5) 0.0043(5) -0.0004(5)
C3 0.0236(6) 0.0164(6) 0.0287(7) -0.0017(5) 0.0027(5) 0.0013(5)
C4 0.0194(6) 0.0229(6) 0.0253(6) -0.0027(5) -0.0033(5) -0.0009(5)
C5 0.0182(6) 0.0152(6) 0.0171(6) -0.0005(4) -0.0008(4) -0.0010(4)
C6 0.0180(6) 0.0173(6) 0.0217(6) 0.0006(5) 0.0005(5) -0.0004(5)
C7 0.0273(7) 0.0214(6) 0.0234(6) 0.0049(5) -0.0050(5) -0.0006(5)
C8 0.0324(7) 0.0238(7) 0.0188(6) 0.0030(5) 0.0000(5) -0.0054(5)
C9 0.0232(6) 0.0240(6) 0.0197(6) -0.0030(5) 0.0048(5) -0.0049(5)
C10 0.0172(6) 0.0168(6) 0.0183(6) -0.0032(5) -0.0009(5) -0.0008(5)
C11 0.0195(6) 0.0259(7) 0.0242(6) -0.0040(5) -0.0003(5) 0.0043(5)
O2 0.0221(5) 0.0413(6) 0.0332(6) -0.0073(5) -0.0054(4) -0.0006(4)
O3 0.0204(5) 0.0279(5) 0.0317(5) 0.0083(4) 0.0053(4) 0.0097(4)
C12 0.0328(8) 0.0480(10) 0.0472(10) 0.0123(8) 0.0052(7) 0.0244(8)
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C5 . C6 . 1.4060(17)    yes
C5 . C10 . 1.4060(17)    yes
C6 . C7 . 1.3940(18)    yes
C6 . O3 . 1.3640(16)    yes
C7 . C8 . 1.388(2)    yes
C7 . H71 . 1.000    no
C8 . C9 . 1.385(2)    yes
C8 . H81 . 1.000    no
C9 . C10 . 1.3948(18)    yes
C9 . H91 . 1.000    no
C10 . C11 . 1.5097(18)    yes
C11 . O2 . 1.4326(17)    yes
C11 . H111 . 1.000    no
C11 . H112 . 1.000    no
O2 . H1 . 1.05(3)    no
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C2 . C1 . C3 . 111.02(11)    yes
S1 . C1 . C4 . 109.52(9)    yes
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C7 . C8 . C9 . 120.88(12)    yes
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_publ_contact_author_address
;
Chemistry Research Laboratory
Oxford  University
Mansfield Rd.
Oxford OX1 3TA
;
_publ_contact_author_phone '44-1865-275642'
_publ_contact_author_fax '44-1865-285002'
_publ_contact_author_email john.brown@chem.ox.ac.uk

_publ_requested_journal 'Organic and Biomolecular Chemistry'


_publ_section_title
;
Sequential ortho-lithiations; the sulfoxide group
as a relay to enable meta-substitution
;
loop_
_publ_author_name
_publ_author_address
'Flemming, Jonathan P.'
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;
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Gunnels Wood Rd.
Stevenage
Herts SG1 2NY
;


_publ_section_abstract
;
Sulfoxides are known to be powerful directing groups for ortho-lithiation,
even in competition with other directors.This has been utilised to
introduce
substituents meta- to a methoxy-group by sequential lithiation,
reaction with Me tert-butylsulfinate, and a second lithiation.
```

```
Electrophilic trapping of the ensuing lithio-compound
with a range of electrophiles followed by reductive removal of the
sulfoxide
led to meta-substituted anisoles.
The X-ray structure of the hydroxymethyl compound is included here.
Some interesting side-reactions were uncovered,including a short synthesis
of quinazolines arising from the use of PhCN in the second step.
;
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data_arc900
#=============================================================
# Diffractometer details
#=============================================================
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;
Nonius KappaCCD
;
_diffrn_radiation_monochromator   graphite
_computing_data_collection
;
KappaCCD software 'Collect' (Nonius, 2000)
;
_computing_data_reduction
;
KappaCCD software 'DENZO' (Otwinowski & Minor, 1996)
;
_computing_cell_refinement
;
KappaCCD software 'DENZO' (Otwinowski & Minor, 1996)
;
#=============================================================
# General computing
#=============================================================
_computing_structure_refinement
;
CRYSTALS (Watkin et al, 2001)
;
_computing_publication_material
;
CRYSTALS (Watkin et al, 2001)
;
_computing_structure_solution     'SIR92 (Giacovazzo et al, 1992)'


_chemical_name_systematic         # IUPAC name, in full
;
?
;
_chemical_melting_point           ?
_refine_ls_matrix_type            full
```

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_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     noref
#=============================================================

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_cell_length_b                    12.0343(4)
_cell_angle_beta                  91.0936(16)
_cell_length_c                    11.7417(4)
_cell_angle_gamma                 90
_cell_volume                      1193.09(7)

_symmetry_cell_setting            'Monoclinic'
_symmetry_space_group_name_H-M    'P 1 21/n 1 '
loop_
 _symmetry_equiv_pos_as_xyz
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 '-x,-y,-z'
 '-x+1/2,y+1/2,-z+1/2'
 'x+1/2,-y+1/2,z+1/2'

loop_
_atom_type_symbol
_atom_type_scat_dispersion_real
_atom_type_scat_dispersion_imag
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_atom_type_scat_Cromer_Mann_b1
_atom_type_scat_Cromer_Mann_a2
_atom_type_scat_Cromer_Mann_b2
_atom_type_scat_Cromer_Mann_a3
_atom_type_scat_Cromer_Mann_b3
_atom_type_scat_Cromer_Mann_a4
_atom_type_scat_Cromer_Mann_b4
_atom_type_scat_Cromer_Mann_c
_atom_type_scat_source
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    1.0200   10.2075    1.5886    0.5687    0.8650   51.6512    0.2156
'International_Tables_Vol_IV_Table_2.2B'
 'H   '    0.0000    0.0000    0.4930   10.5109
    0.3229   26.1257    0.1402    3.1424    0.0408   57.7998    0.0030
'International_Tables_Vol_IV_Table_2.2B'
 'O   '    0.0106    0.0060    3.0485   13.2771
    2.2868    5.7011    1.5463    0.3239    0.8670   32.9089    0.2508
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 'S   '    0.1246    0.1234    6.9053    1.4679
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'International_Tables_Vol_IV_Table_2.2B'
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_chemical_compound_source
;
synthesis as described
;
_chemical_formula_weight            226.34


_cell_measurement_reflns_used         8955
_cell_measurement_theta_min              5
_cell_measurement_theta_max             28
_cell_measurement_temperature          150

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_exptl_crystal_colour             ' colourless '
_exptl_crystal_size_min           0.06
_exptl_crystal_size_mid           0.28
_exptl_crystal_size_max           0.28

_exptl_crystal_density_diffrn     1.260
_exptl_crystal_density_meas       ?
# Non-dispersive F(000):
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_exptl_absorpt_coefficient_mu     0.250

_diffrn_measurement_method        \w

_exptl_absorpt_correction_type    multi-scan
_exptl_absorpt_process_details
;
 Denzo/Scalepack (Otwinowski & Minor, 1996)
;
_exptl_absorpt_correction_T_min   0.93
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_diffrn_standards_decay_%         0.00

_diffrn_ambient_temperature        150
_diffrn_reflns_number             8955
_reflns_number_total              2702
#2830 unique reflcetions including absences
_diffrn_reflns_av_R_equivalents   0.027

_diffrn_reflns_theta_min          5.118
_diffrn_reflns_theta_max          27.479
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_diffrn_reflns_theta_full         27.479
_diffrn_measured_fraction_theta_full 0.994


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_diffrn_reflns_limit_h_max        10
_diffrn_reflns_limit_k_min        -15
_diffrn_reflns_limit_k_max        15
_diffrn_reflns_limit_l_min        -15
_diffrn_reflns_limit_l_max        15
_reflns_limit_h_min               -10
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_refine_diff_density_max          0.25


_refine_ls_number_reflns          2132
_refine_ls_number_restraints      0
_refine_ls_number_parameters      136

#_refine_ls_R_factor_ref 0.0326
_refine_ls_wR_factor_ref          0.0442
_refine_ls_goodness_of_fit_ref    0.9915

#_reflns_number_all 2702
_refine_ls_R_factor_all           0.0446
_refine_ls_wR_factor_all          0.0494

# The I/u(I) cutoff below was used for refinement as
# well as the _gt R-factors:
_reflns_threshold_expression      I>3.00u(I)
_reflns_number_gt                 2132
_refine_ls_R_factor_gt            0.0326
_refine_ls_wR_factor_gt           0.0442


_refine_ls_shift/su_max           0.000394
_refine_ls_structure_factor_coef  F
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
;
 Method, part 1, Chebychev polynomial, (Watkin, 1994, Prince, 1982)
```

```
 [weight] = 1.0/[A~0~*T~0~(x)+A~1~*T~1~(x) ... +A~n-1~]*T~n-1~(x)]
 where A~i~ are the Chebychev coefficients listed below and x= Fcalc/Fmax
 Method = Robust Weighting (Prince, 1982)
 W = [weight] * [1-(deltaF/6*sigmaF)^2^]^2^
 A~i~ are:
 1.09 0.537 0.709
;
_diffrn_radiation_type            'Mo K\a'
_diffrn_radiation_wavelength           0.71073

# Uequiv = arithmetic mean of Ui
# i.e. Ueqiv = (U1+U2+U3)/3

loop_
_atom_site_label
_atom_site_type_symbol
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_U_iso_or_equiv
_atom_site_occupancy
_atom_site_adp_type
S1 S 0.31522(4) 0.37442(3) 0.23368(3) 0.0198 1.0000 Uani
O1 O 0.16389(14) 0.40533(11) 0.17331(10) 0.0319 1.0000 Uani
C1 C 0.41494(17) 0.50846(11) 0.26956(12) 0.0193 1.0000 Uani
C2 C 0.56466(18) 0.48595(13) 0.34059(13) 0.0241 1.0000 Uani
C3 C 0.4567(2) 0.55538(14) 0.15272(14) 0.0288 1.0000 Uani
C4 C 0.30217(19) 0.58581(13) 0.33128(14) 0.0254 1.0000 Uani
C5 C 0.26987(17) 0.32239(11) 0.37364(12) 0.0189 1.0000 Uani
C6 C 0.38514(17) 0.25116(12) 0.42180(12) 0.0207 1.0000 Uani
C7 C 0.36517(19) 0.20477(13) 0.52934(13) 0.0253 1.0000 Uani
C8 C 0.2259(2) 0.22724(13) 0.58618(13) 0.0276 1.0000 Uani
C9 C 0.10773(19) 0.29211(13) 0.53662(14) 0.0269 1.0000 Uani
C10 C 0.12672(18) 0.34037(12) 0.42931(13) 0.0226 1.0000 Uani
C11 C -0.0136(2) 0.40227(16) 0.37881(16) 0.0341 1.0000 Uani
O3 O 0.51492(13) 0.23179(10) 0.35699(10) 0.0283 1.0000 Uani
C12 C 0.6132(2) 0.13927(17) 0.38786(17) 0.0402 1.0000 Uani
H21 H 0.6179 0.5580 0.3597 0.0289 1.0000 Uiso
H22 H 0.5360 0.4469 0.4125 0.0289 1.0000 Uiso
H23 H 0.6382 0.4383 0.2961 0.0289 1.0000 Uiso
H31 H 0.5116 0.6285 0.1627 0.0346 1.0000 Uiso
H32 H 0.5283 0.5023 0.1131 0.0346 1.0000 Uiso
H33 H 0.3576 0.5660 0.1060 0.0346 1.0000 Uiso
H41 H 0.3576 0.6571 0.3498 0.0305 1.0000 Uiso
H42 H 0.2673 0.5497 0.4033 0.0305 1.0000 Uiso
H43 H 0.2076 0.6013 0.2812 0.0305 1.0000 Uiso
H71 H 0.4490 0.1565 0.5647 0.0303 1.0000 Uiso
H81 H 0.2107 0.1961 0.6642 0.0331 1.0000 Uiso
H91 H 0.0071 0.3046 0.5783 0.0324 1.0000 Uiso
H111 H -0.1010 0.4039 0.4351 0.0410 1.0000 Uiso
```

```
H112 H 0.0185 0.4801 0.3602 0.0410 1.0000 Uiso
H113 H -0.0512 0.3638 0.3078 0.0410 1.0000 Uiso
H121 H 0.7034 0.1342 0.3342 0.0482 1.0000 Uiso
H122 H 0.6554 0.1497 0.4673 0.0482 1.0000 Uiso
H123 H 0.5495 0.0693 0.3837 0.0482 1.0000 Uiso
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_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
S1 0.02379(19) 0.01964(18) 0.01577(18) -0.00108(12) -0.00170(12) -0.00046
(13)
O1 0.0298(6) 0.0375(6) 0.0279(6) 0.0034(5) -0.0143(5) -0.0041(5)
C1 0.0200(6) 0.0184(6) 0.0194(6) 0.0008(5) -0.0007(5) 0.0006(5)
C2 0.0239(7) 0.0228(7) 0.0257(7) -0.0022(6) -0.0035(5) -0.0004(6)
C3 0.0331(8) 0.0283(7) 0.0252(8) 0.0082(6) 0.0039(6) -0.0001(6)
C4 0.0269(7) 0.0205(7) 0.0289(8) -0.0017(6) 0.0013(6) 0.0035(6)
C5 0.0212(7) 0.0167(6) 0.0186(6) -0.0008(5) -0.0003(5) -0.0006(5)
C6 0.0205(6) 0.0182(6) 0.0233(7) -0.0002(5) 0.0006(5) -0.0011(5)
C7 0.0302(8) 0.0211(7) 0.0244(7) 0.0038(6) -0.0034(6) -0.0012(6)
C8 0.0379(9) 0.0239(7) 0.0210(7) 0.0020(6) 0.0022(6) -0.0062(6)
C9 0.0288(8) 0.0247(7) 0.0275(8) -0.0022(6) 0.0075(6) -0.0046(6)
C10 0.0219(7) 0.0200(7) 0.0259(7) -0.0020(5) 0.0015(5) -0.0008(5)
C11 0.0258(8) 0.0380(9) 0.0386(9) -0.0007(7) 0.0028(7) 0.0053(7)
O3 0.0244(5) 0.0273(6) 0.0334(6) 0.0074(4) 0.0062(4) 0.0091(4)
C12 0.0376(10) 0.0427(10) 0.0403(10) 0.0074(8) 0.0027(8) 0.0221(8)
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S1 . O1 . 1.4965(11)    yes
S1 . C1 . 1.8642(14)    yes
S1 . C5 . 1.8065(14)    yes
C1 . C2 . 1.5255(19)    yes
C1 . C3 . 1.531(2)    yes
C1 . C4 . 1.525(2)    yes
C2 . H21 . 1.000    no
C2 . H22 . 1.000    no
C2 . H23 . 1.000    no
C3 . H31 . 1.000    no
C3 . H32 . 1.000    no
C3 . H33 . 1.000    no
```

```
C4 . H41 . 1.000    no
C4 . H42 . 1.000    no
C4 . H43 . 1.000    no
C5 . C6 . 1.407(2)    yes
C5 . C10 . 1.402(2)    yes
C6 . C7 . 1.394(2)    yes
C6 . O3 . 1.3662(18)    yes
C7 . C8 . 1.390(2)    yes
C7 . H71 . 1.000    no
C8 . C9 . 1.386(2)    yes
C8 . H81 . 1.000    no
C9 . C10 . 1.399(2)    yes
C9 . H91 . 1.000    no
C10 . C11 . 1.512(2)    yes
C11 . H111 . 1.000    no
C11 . H112 . 1.000    no
C11 . H113 . 1.000    no
O3 . C12 . 1.431(2)    yes
C12 . H121 . 1.000    no
C12 . H122 . 1.000    no
C12 . H123 . 1.000    no
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O1 . S1 . C1 . 105.66(7)    yes
O1 . S1 . C5 . 108.85(7)    yes
C1 . S1 . C5 . 101.36(6)    yes
S1 . C1 . C2 . 109.64(10)    yes
S1 . C1 . C3 . 103.15(10)    yes
C2 . C1 . C3 . 110.64(12)    yes
S1 . C1 . C4 . 110.61(10)    yes
C2 . C1 . C4 . 111.55(12)    yes
C3 . C1 . C4 . 110.95(12)    yes
C1 . C2 . H21 . 109.467    no
C1 . C2 . H22 . 109.467    no
H21 . C2 . H22 . 109.476    no
C1 . C2 . H23 . 109.467    no
H21 . C2 . H23 . 109.476    no
H22 . C2 . H23 . 109.476    no
C1 . C3 . H31 . 109.467    no
C1 . C3 . H32 . 109.467    no
H31 . C3 . H32 . 109.476    no
C1 . C3 . H33 . 109.467    no
H31 . C3 . H33 . 109.476    no
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H32 . C3 . H33 . 109.476    no
C1 . C4 . H41 . 109.467    no
C1 . C4 . H42 . 109.467    no
H41 . C4 . H42 . 109.476    no
C1 . C4 . H43 . 109.467    no
H41 . C4 . H43 . 109.476    no
H42 . C4 . H43 . 109.476    no
S1 . C5 . C6 . 114.81(10)    yes
S1 . C5 . C10 . 124.77(11)    yes
C6 . C5 . C10 . 120.17(13)    yes
C5 . C6 . C7 . 120.88(14)    yes
C5 . C6 . O3 . 115.86(13)    yes
C7 . C6 . O3 . 123.26(13)    yes
C6 . C7 . C8 . 118.37(14)    yes
C6 . C7 . H71 . 120.814    no
C8 . C7 . H71 . 120.814    no
C7 . C8 . C9 . 121.13(14)    yes
C7 . C8 . H81 . 119.434    no
C9 . C8 . H81 . 119.434    no
C8 . C9 . C10 . 121.18(14)    yes
C8 . C9 . H91 . 119.411    no
C10 . C9 . H91 . 119.411    no
C5 . C10 . C9 . 118.06(14)    yes
C5 . C10 . C11 . 124.74(14)    yes
C9 . C10 . C11 . 117.08(14)    yes
C10 . C11 . H111 . 109.467    no
C10 . C11 . H112 . 109.467    no
H111 . C11 . H112 . 109.476    no
C10 . C11 . H113 . 109.467    no
H111 . C11 . H113 . 109.476    no
H112 . C11 . H113 . 109.476    no
C6 . O3 . C12 . 117.34(13)    yes
O3 . C12 . H121 . 109.467    no
O3 . C12 . H122 . 109.467    no
H121 . C12 . H122 . 109.476    no
O3 . C12 . H123 . 109.467    no
H121 . C12 . H123 . 109.476    no
H122 . C12 . H123 . 109.476    no
```