Electronic Supplementary Material (ESI) for RSC Advances

Supplementary Information

Thermal Fragmentation of Spirodithiohydantoins: A Novel Route to NHCs

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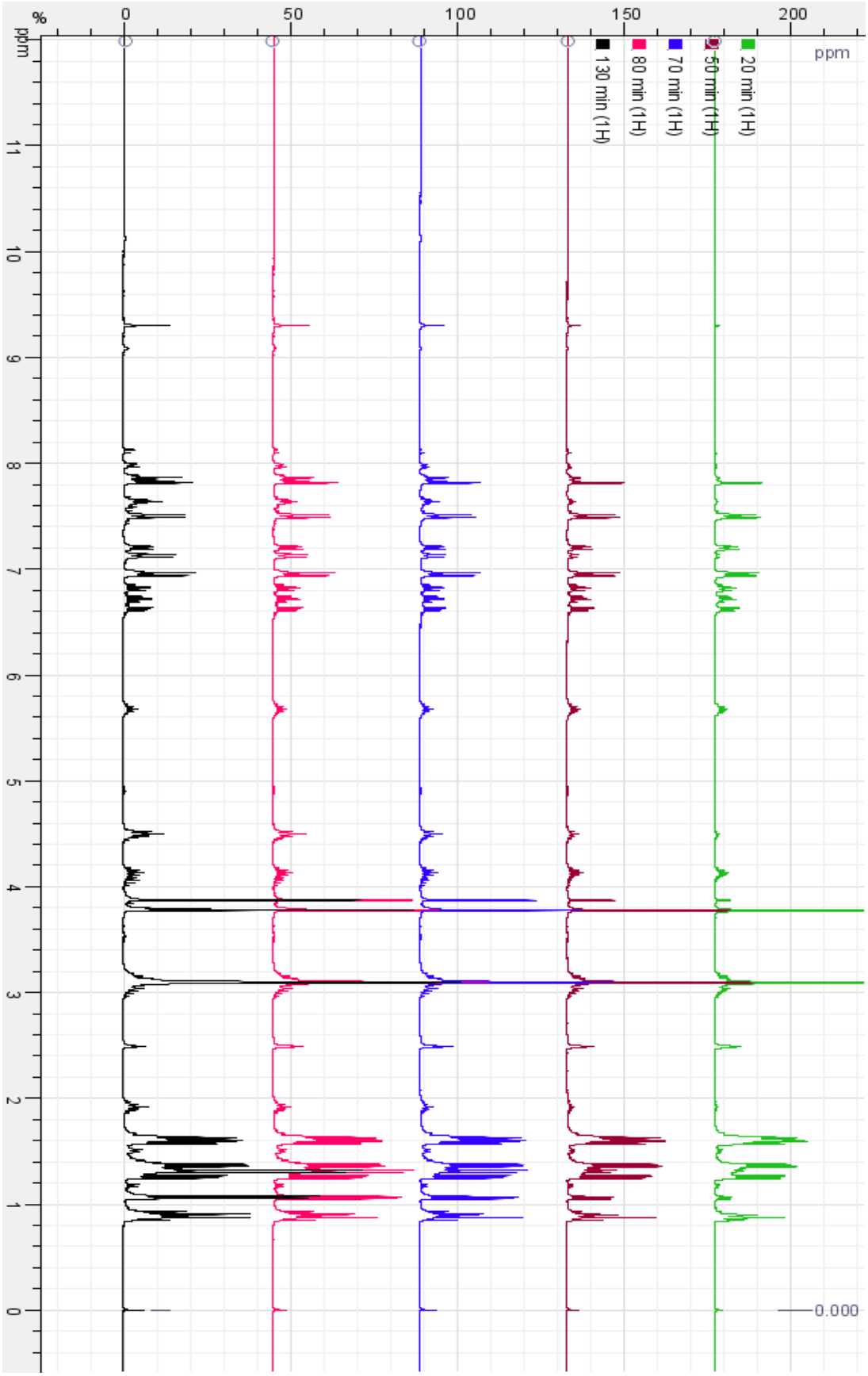
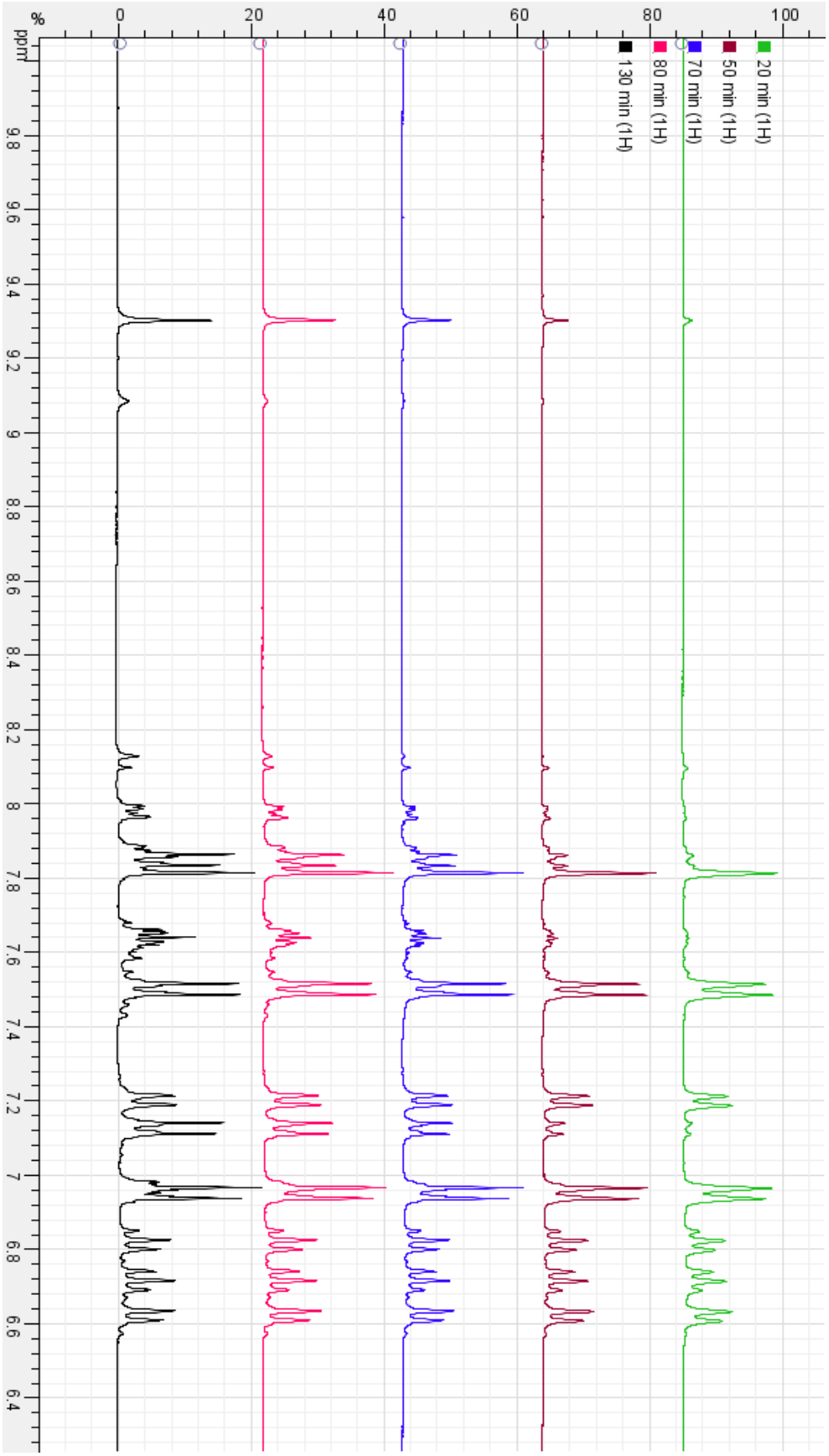
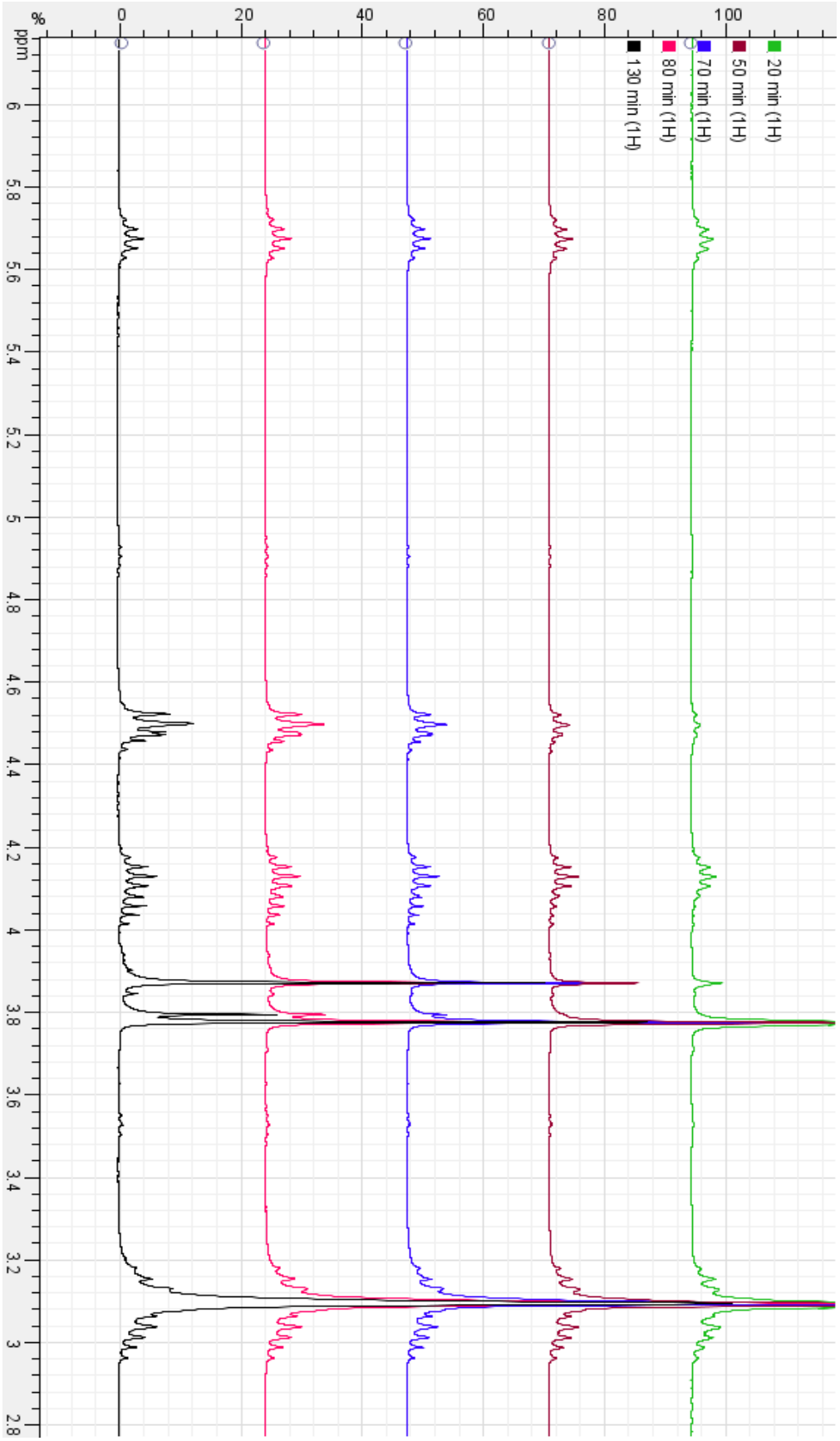
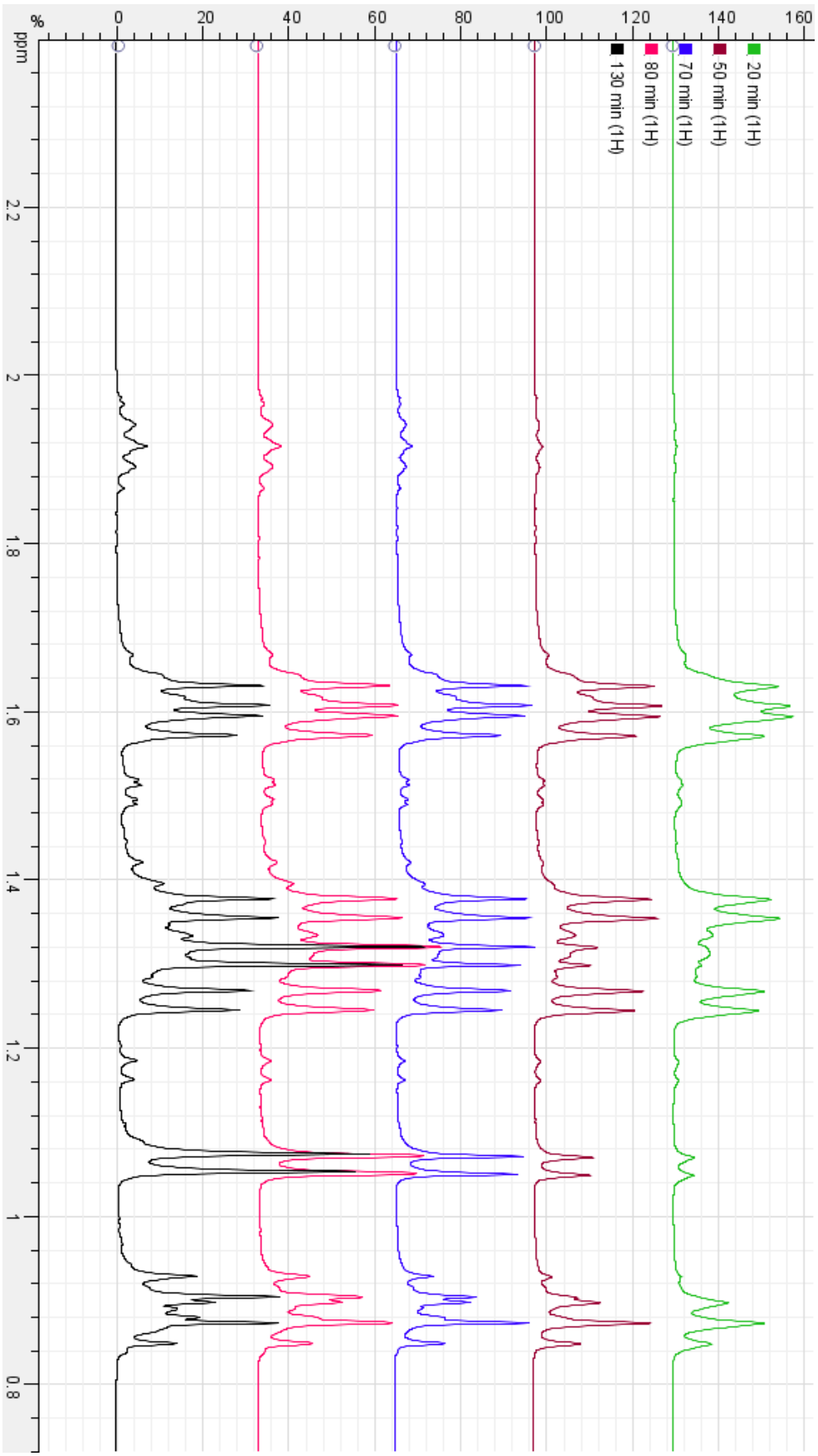
Table of Contents









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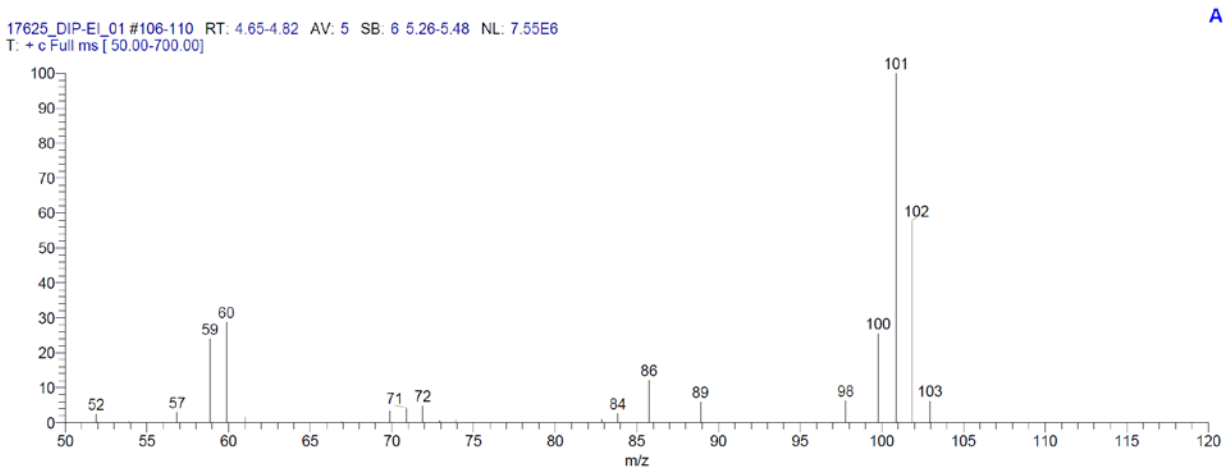
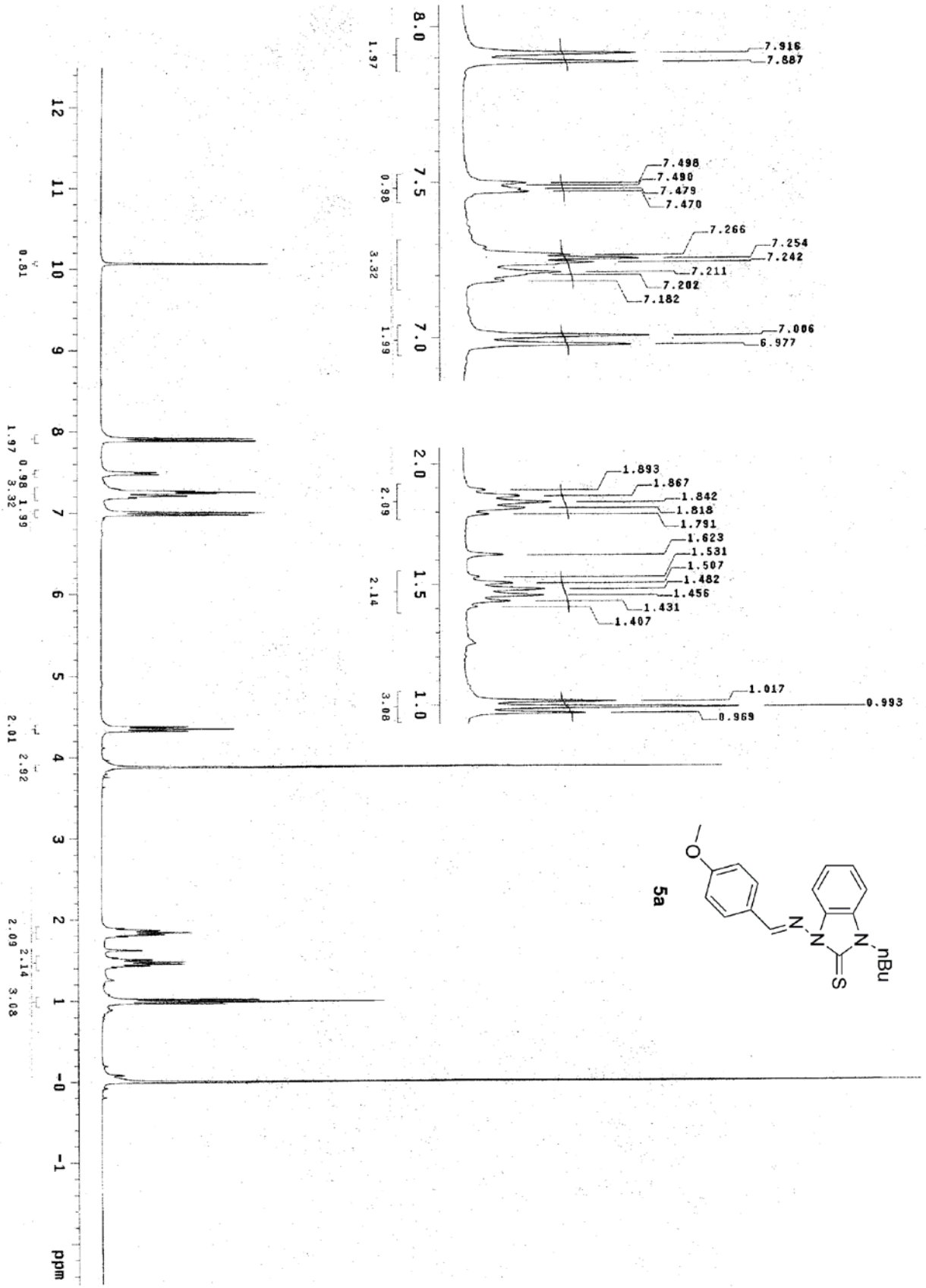
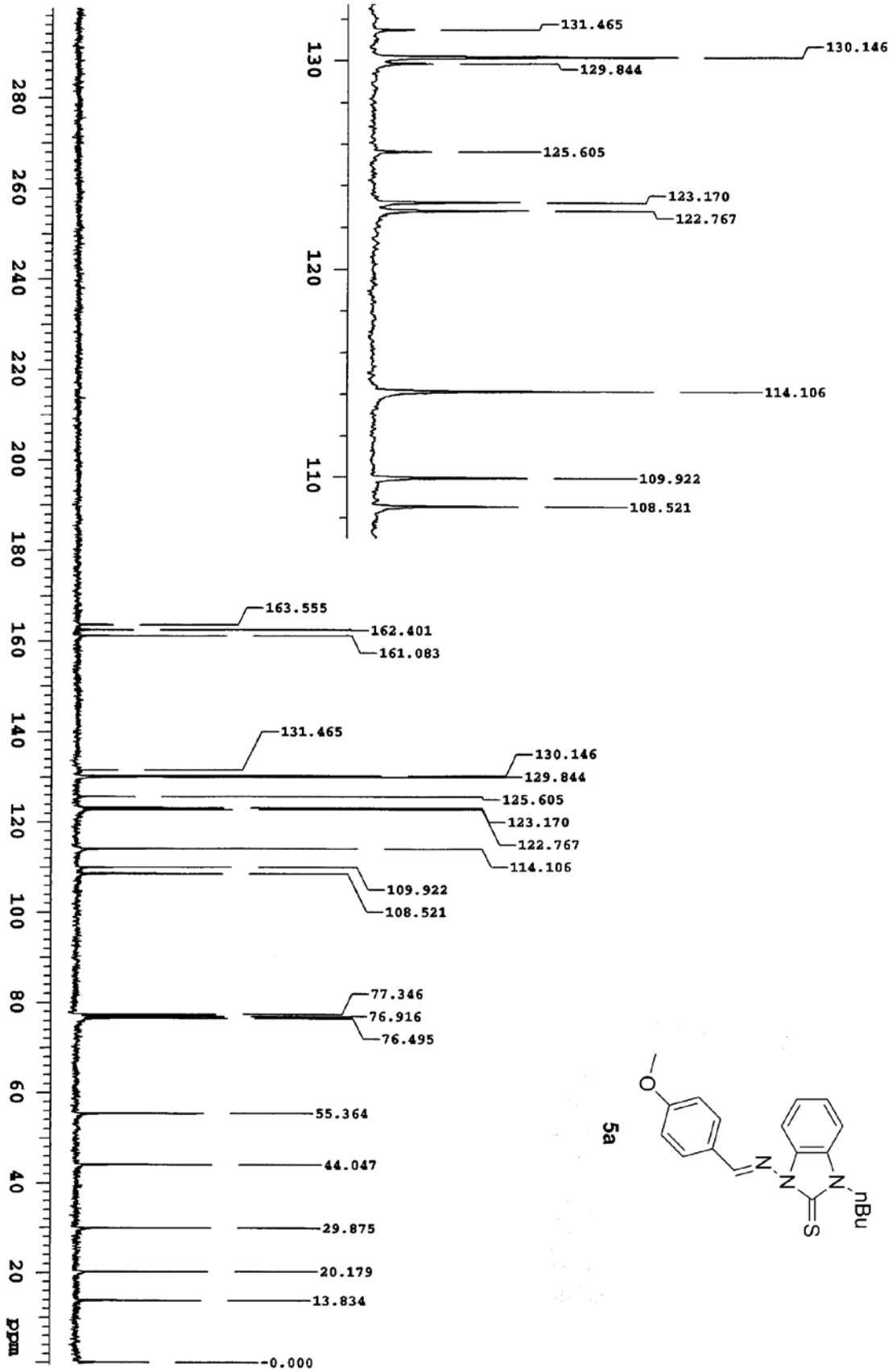
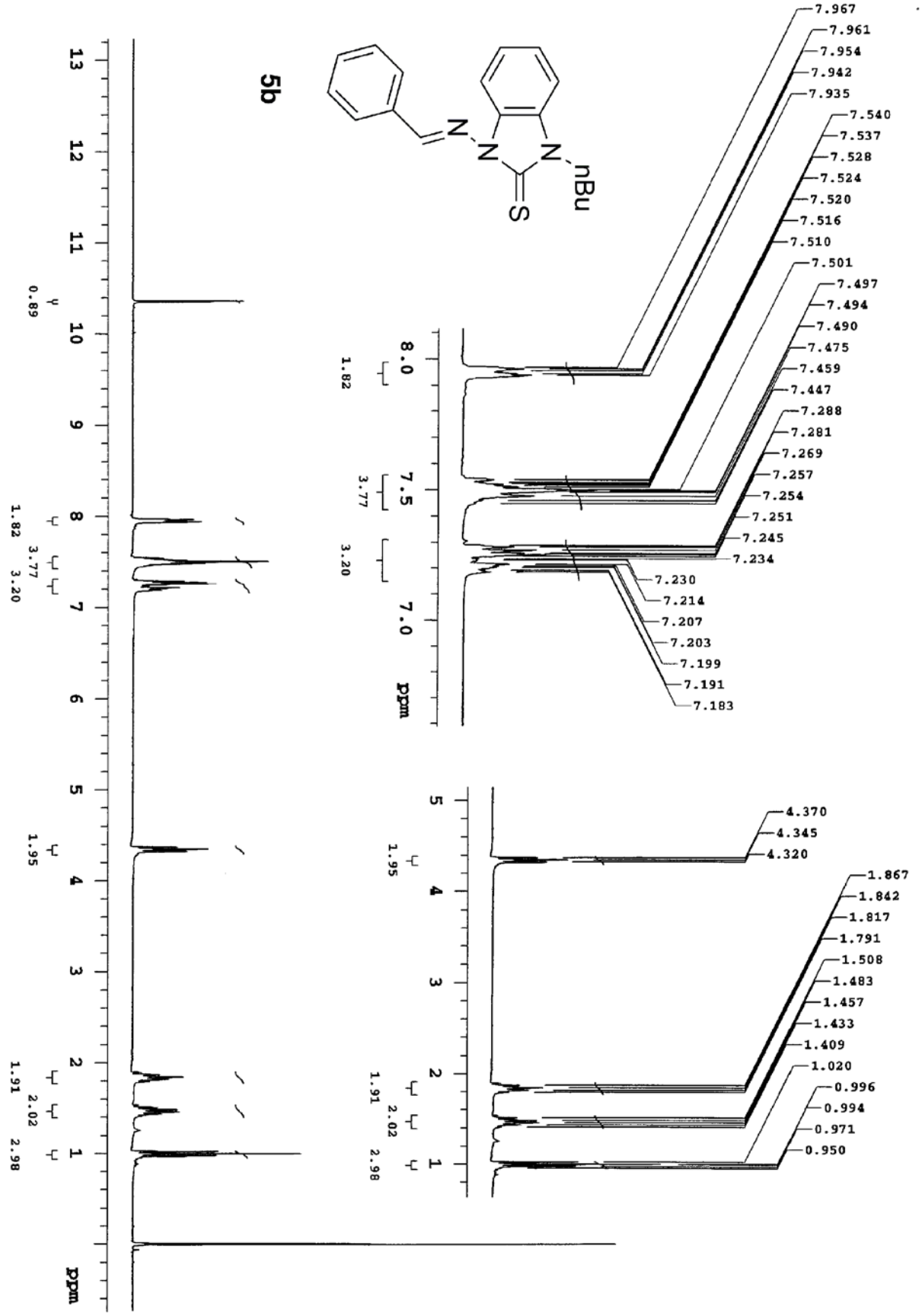
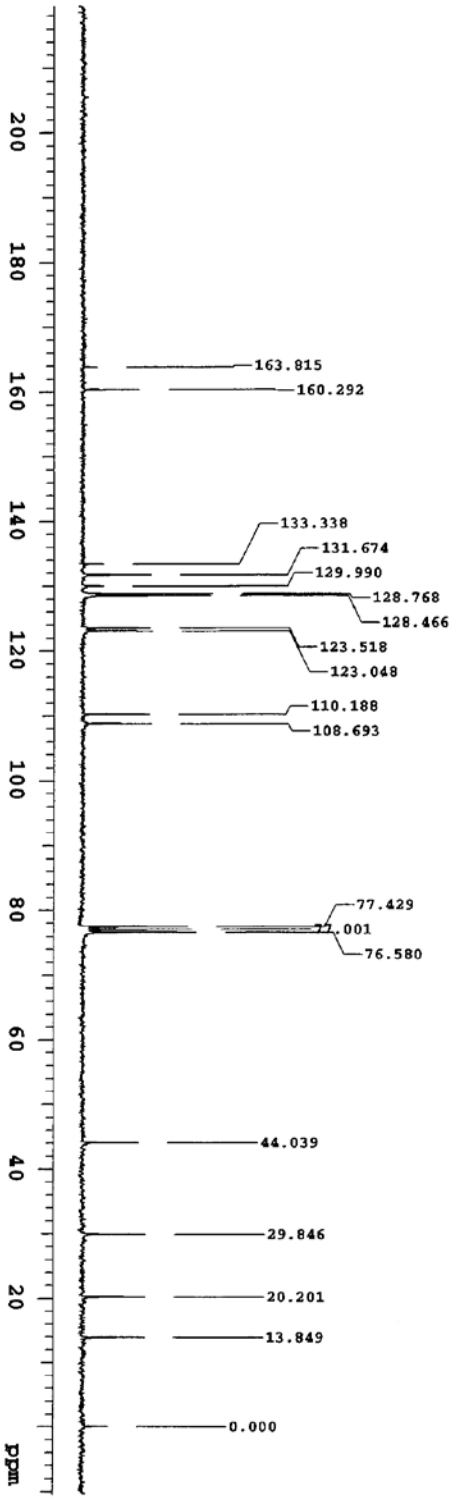


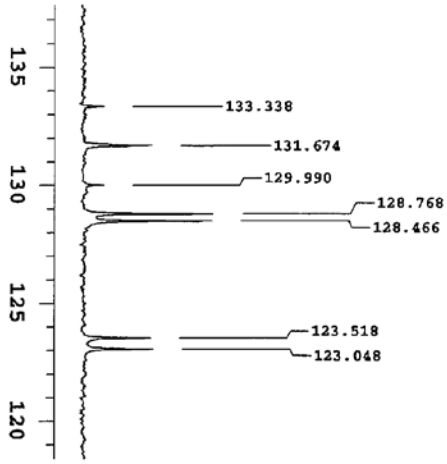


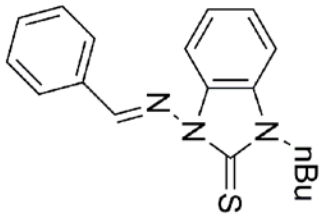






INDEX	FREQUENCY	PPM	HEIGHT
1	12360.197	163.815	2.4
2	12094.329	160.292	9.5
3	10060.600	133.338	3.5
4	9935.081	131.674	11.5
5	9807.973	129.990	3.4
6	9715.820	128.768	21.9
7	9693.046	128.466	21.6
8	9319.667	123.518	11.3
9	9284.182	123.048	11.8
10	8313.924	110.188	11.2
11	8201.116	108.693	11.6
12	5842.203	77.429	17.7
13	5809.896	77.001	18.4
14	5778.119	76.580	19.1
15	3322.816	44.039	9.5
16	2251.931	29.846	10.7
17	1524.237	20.201	10.6
18	1044.934	13.849	10.5
19	0.000	0.000	4.4





5b

Computing details

Data collection: 'Mon Jul 30 16:40:32 2012'; cell refinement: 'Mon Jul 30 16:40:32 2012'; data reduction: 'Mon Jul 30 16:40:32 2012'; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008).

References

NOT FOUND

(d469)

Crystal data

$C_{18}H_{19}N_3S$	$F(000) = 656$
$M_r = 309.42$	$D_x = 1.280$ Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Hall symbol: -P 2ybc	Cell parameters from 4832 reflections
$a = 5.86514$ (13) Å	$\theta = 3.0–73.7°$
$b = 14.6361$ (3) Å	$\mu = 1.77$ mm^{-1}
$c = 18.7566$ (3) Å	$T = 120$ K
$\beta = 94.0394$ (18)°	Block, colorless
$V = 1606.12$ (5) Å^3	0.50 × 0.44 × 0.15 mm
$Z = 4$	

Data collection

SuperNova, Dual, Cu at zero, Atlas diffractometer	2883 independent reflections
Radiation source: fine-focus sealed tube	2446 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.030$
Detector resolution: 10.6501 pixels mm^{-1}	$\theta_{max} = 67.5°$, $\theta_{min} = 3.8°$
ω scans	$h = -5\rightarrow7$
Absorption correction: multi-scan 'Mon Jul 30 16:40:32 2012'	$k = -17\rightarrow17$
$T_{min} = 0.629$, $T_{max} = 1.000$	$l = -22\rightarrow22$
10315 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.034$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.094$	H-atom parameters constrained
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.655P]$ where $P = (F_o^2 + 2F_c^2)/3$
2883 reflections	$(\Delta/\sigma)_{max} = 0.001$
199 parameters	$\Delta\rangle_{max} = 0.21$ e Å^{-3}
0 restraints	$\Delta\rangle_{min} = -0.20$ e Å^{-3}

Special details

Geometry. 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.

Refinement. 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 $F^2 > 2\text{sigma}(F^2)$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	-0.0317 (2)	0.85784 (9)	0.29438 (7)	0.0203 (3)
C2	0.1103 (3)	0.81466 (10)	0.34651 (8)	0.0214 (3)
S2	0.08430 (8)	0.80657 (3)	0.43433 (2)	0.02832 (14)
N3	0.2790 (2)	0.77503 (9)	0.31040 (7)	0.0209 (3)
C4	0.3720 (3)	0.76616 (11)	0.18065 (8)	0.0231 (4)
H4A	0.5046	0.7314	0.1875	0.028*
C5	0.2912 (3)	0.79587 (11)	0.11318 (9)	0.0246 (4)
H5A	0.3717	0.7806	0.0739	0.030*

C6	0.0932 (3)	0.84788 (11)	0.10281 (8)	0.0246 (4)
H6A	0.0445	0.8663	0.0568	0.030*
C7	-0.0341 (3)	0.87311 (11)	0.15962 (8)	0.0232 (4)
H7A	-0.1662	0.9081	0.1529	0.028*
C8	0.0476 (3)	0.84304 (10)	0.22678 (8)	0.0198 (3)
C9	0.2448 (3)	0.79094 (10)	0.23725 (8)	0.0200 (3)
C10	0.4627 (3)	0.71937 (11)	0.34413 (9)	0.0234 (4)
H10A	0.5044	0.7431	0.3916	0.028*
H10B	0.5958	0.7237	0.3164	0.028*
C11	0.3935 (3)	0.61947 (11)	0.34994 (9)	0.0257 (4)
H11A	0.2640	0.6150	0.3792	0.031*
H11B	0.3460	0.5965	0.3027	0.031*
C12	0.5879 (3)	0.56046 (12)	0.38251 (10)	0.0317 (4)
H12A	0.5389	0.4972	0.3817	0.038*
H12B	0.7171	0.5653	0.3531	0.038*
C13	0.6653 (4)	0.58666 (13)	0.45894 (11)	0.0446 (5)
H13A	0.7876	0.5471	0.4763	0.067*
H13B	0.5395	0.5806	0.4887	0.067*
H13C	0.7177	0.6488	0.4601	0.067*
N2	-0.2307 (2)	0.90894 (9)	0.29860 (7)	0.0218 (3)
C14	-0.2561 (3)	0.95293 (11)	0.35664 (8)	0.0224 (3)
H14A	-0.1469	0.9484	0.3949	0.027*
C15	-0.4584 (3)	1.01023 (10)	0.36212 (8)	0.0223 (4)
C16	-0.6298 (3)	1.01697 (11)	0.30652 (9)	0.0252 (4)
H16A	-0.6179	0.9838	0.2647	0.030*
C17	-0.8164 (3)	1.07284 (12)	0.31377 (11)	0.0318 (4)
H17A	-0.9285	1.0779	0.2764	0.038*
C18	-0.8377 (3)	1.12156 (12)	0.37664 (11)	0.0356 (5)
H18A	-0.9651	1.1582	0.3816	0.043*
C19	-0.6697 (3)	1.11537 (12)	0.43156 (10)	0.0333 (4)
H19A	-0.6840	1.1479	0.4736	0.040*
C20	-0.4792 (3)	1.06074 (11)	0.42449 (9)	0.0276 (4)
H20A	-0.3650	1.0578	0.4614	0.033*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0209 (7)	0.0210 (7)	0.0191 (6)	0.0026 (5)	0.0028 (5)	0.0000 (5)
C2	0.0231 (9)	0.0187 (8)	0.0222 (8)	-0.0004 (6)	0.0005 (6)	0.0008 (6)
S2	0.0349 (3)	0.0315 (2)	0.0189 (2)	0.00585 (18)	0.00411 (17)	0.00303 (15)
N3	0.0219 (7)	0.0207 (7)	0.0198 (7)	0.0027 (5)	0.0008 (5)	0.0011 (5)
C4	0.0230 (9)	0.0200 (8)	0.0268 (8)	0.0006 (7)	0.0039 (7)	-0.0018 (6)
C5	0.0297 (10)	0.0221 (8)	0.0228 (8)	-0.0034 (7)	0.0077 (7)	-0.0028 (6)
C6	0.0310 (9)	0.0226 (8)	0.0199 (8)	-0.0028 (7)	-0.0007 (7)	0.0007 (6)
C7	0.0248 (9)	0.0203 (8)	0.0242 (8)	0.0012 (7)	-0.0011 (7)	0.0008 (6)
C8	0.0202 (8)	0.0170 (7)	0.0222 (8)	-0.0014 (6)	0.0024 (6)	-0.0015 (6)
C9	0.0225 (9)	0.0169 (7)	0.0203 (8)	-0.0021 (6)	0.0006 (6)	0.0007 (6)
C10	0.0218 (9)	0.0236 (8)	0.0244 (8)	0.0028 (7)	-0.0016 (7)	0.0020 (6)
C11	0.0268 (9)	0.0222 (8)	0.0275 (8)	0.0019 (7)	-0.0028 (7)	-0.0014 (7)
C12	0.0338 (10)	0.0225 (8)	0.0373 (10)	0.0030 (8)	-0.0081 (8)	0.0018 (7)
C13	0.0625 (14)	0.0275 (10)	0.0400 (11)	-0.0002 (10)	-0.0225 (10)	0.0057 (8)
N2	0.0199 (7)	0.0201 (6)	0.0257 (7)	0.0025 (5)	0.0039 (6)	0.0011 (5)
C14	0.0239 (9)	0.0204 (8)	0.0231 (8)	-0.0012 (7)	0.0031 (7)	0.0025 (6)
C15	0.0245 (9)	0.0176 (7)	0.0257 (8)	-0.0019 (7)	0.0085 (7)	0.0029 (6)
C16	0.0237 (9)	0.0198 (8)	0.0326 (9)	-0.0029 (7)	0.0055 (7)	0.0004 (7)
C17	0.0226 (9)	0.0246 (9)	0.0484 (11)	-0.0014 (7)	0.0045 (8)	0.0047 (8)
C18	0.0288 (10)	0.0205 (8)	0.0600 (12)	0.0027 (7)	0.0207 (9)	0.0029 (8)
C19	0.0418 (12)	0.0222 (8)	0.0386 (10)	-0.0002 (8)	0.0218 (9)	-0.0018 (7)
C20	0.0357 (10)	0.0225 (8)	0.0259 (9)	-0.0011 (7)	0.0108 (7)	0.0027 (7)

Geometric parameters (Å, º)

N1—C2	1.391 (2)	C8—C9	1.388 (2)
N1—N2	1.3929 (18)	C10—C11	1.523 (2)
N1—C8	1.3979 (19)	C11—C12	1.523 (2)
C2—N3	1.367 (2)	C12—C13	1.522 (2)
C2—S2	1.6692 (16)	N2—C14	1.283 (2)
N3—C9	1.3922 (19)	C14—C15	1.463 (2)
N3—C10	1.459 (2)	C15—C20	1.397 (2)

C4—C9	1.388 (2)	C15—C16	1.400 (2)
C4—C5	1.389 (2)	C16—C17	1.381 (2)
C5—C6	1.391 (2)	C17—C18	1.391 (3)
C6—C7	1.393 (2)	C18—C19	1.377 (3)
C7—C8	1.387 (2)	C19—C20	1.388 (3)
C2—N1—N2	131.81 (13)	C8—C9—C4	121.52 (15)
C2—N1—C8	110.26 (13)	C8—C9—N3	106.98 (13)
N2—N1—C8	117.92 (12)	C4—C9—N3	131.48 (15)
N3—C2—N1	105.25 (13)	N3—C10—C11	112.01 (14)
N3—C2—S2	125.33 (12)	C12—C11—C10	112.17 (14)
N1—C2—S2	129.37 (12)	C13—C12—C11	113.59 (15)
C2—N3—C9	111.00 (13)	C14—N2—N1	117.76 (13)
C2—N3—C10	124.08 (13)	N2—C14—C15	119.40 (15)
C9—N3—C10	124.84 (13)	C20—C15—C16	119.10 (16)
C9—C4—C5	116.59 (15)	C20—C15—C14	118.79 (16)
C4—C5—C6	121.78 (15)	C16—C15—C14	122.11 (15)
C5—C6—C7	121.68 (15)	C17—C16—C15	120.07 (16)
C8—C7—C6	116.17 (15)	C16—C17—C18	120.40 (18)
C7—C8—C9	122.27 (15)	C19—C18—C17	119.88 (17)
C7—C8—N1	131.20 (15)	C18—C19—C20	120.30 (17)
C9—C8—N1	106.50 (13)	C19—C20—C15	120.23 (17)