Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting Information

Iodination of anilines and phenols with 18-crown-6 supported ICl_2^-

Hannah W. Mbatia,[a] Olbelina Ulloa,[a] Daniel P. Kennedy,[a] Christopher D. Incarvito[b] and Shawn C. Burdette [a,*]

[a] *Department of Chemistry, University of Connecticut, Storrs, CT 06269-3060, USA. E-mail: shawn.burdette@uconn.edu*

[b] *Department of Chemistry, Yale University, 225 Prospect Street, P. O. Box 208107, New Haven, CT 06520-8107, USA*

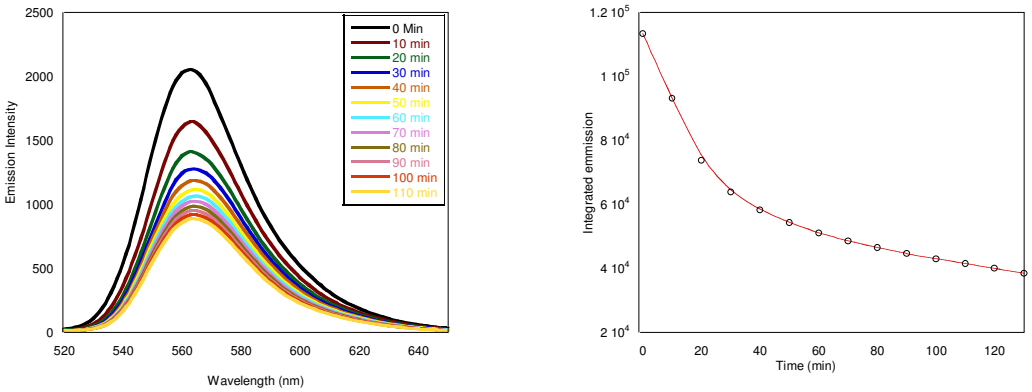


Fig S-1 Changes in fluorescence emission at 5µM 2, 7-dichloroflourescein in CH_3CN after the addition of 30µM $\{[K.18\text{-}C\text{-}6]ICl_2\}_n$ and 10µM Et_3N (left). Fluorescence response versus time obtained by integrating the emission spectra between 520-650 nm (right).

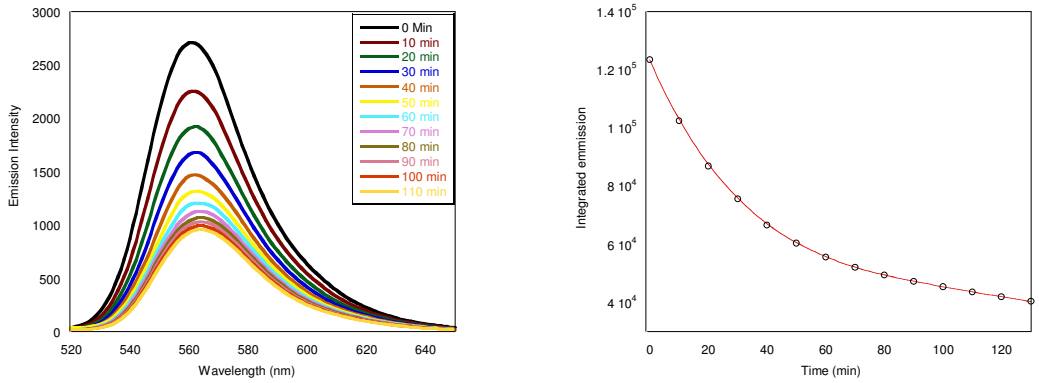


Fig S-2 Changes in fluorescence emission at 10µM 2, 7-dichloroflourescein in CH_3CN after the addition of 15µM {[K.18-C-6]ICl_2} and 10µM Et_3N (left). Fluorescence response versus time obtained by integrating the emission spectra between 520-650 nm (right).

Crystallographic information

Data Collection

A yellow block crystal of $IKCl_2O_6C_{12}H_{24}$ having approximate dimensions of 0.25 x 0.10 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku Mercury2 CCD area detector with graphite monochromated Mo-Kα radiation.Indexing was performed from 6 images that were exposed for 10.0 seconds. The crystal-to-detector distance was 49.90 mm.Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 8.8860(9) Å

b = 8.1987(8) Å

β = 101.873(2)^o

c = 13.9274(13) Å

V = 992.96(16) Å^3

For Z = 2 and F.W. = 501.23, the calculated density is 1.676 g/cm^3. The systematic absences of:

h0l: h+l ± 2n

0k0: k ± 2n

Uniquely determine the space group to be:

$P2_1/n$ (#14)

The data were collected at a temperature of -50 ± 1^{o}C to a maximum 2θ value of 55.0^o. A total of 540 oscillation images were collected. A sweep of data was done using ω scans from -120.0 to 60.0^o in 1.0^o step, at χ=54.0^o and ϕ = 0.0^o. The exposure rate was 30.0 [sec./^{o}]. The detector swing angle was -28.40^o. A second sweep was performed using ω scans from -120.0 to 60.0^o in 1.0^o step, at χ=54.0^o and ϕ = 120.0^o. The exposure rate was 30.0 [sec./^{o}]. The detector swing angle was -28.40^o. Another sweep was performed using ω scans from -120.0 to 60.0^o in 1.0^o step, at χ=54.0^o and ϕ = 240.0^o. The exposure rate was 30.0 [sec./^{o}]. The detector swing angle was -28.40^o. The crystal-to-detector distance was 49.90 mm. Readout was performed in the 0.146 mm pixel mode.

Data Reduction Of the 10037 reflections that were collected, 2272 were unique (R_{int} = 0.035). Data were collected and processed using CrystalClear (Rigaku). Net intensities and sigmas were derived as follows:

$$F^2 = [\Sigma(P_i - mB_{ave})] \cdot Lp^{-1}$$

where P_i is the value in counts of the i^{th} pixel

m is the number of pixels in the integration area

B_{ave} is the background average

Lp is the Lorentz and polarization factor

$$B_{ave} = \Sigma(B_j)/n$$

where n is the number of pixels in the background area

B_j is the value of the j^{th} pixel in counts

$$\sigma^2(F^2_{hkl}) = [(\Sigma P_i) + m((\Sigma(B_{ave} - B_j)^2)/(n-1))] \cdot Lp \cdot errmul + (erradd \cdot F^2)^2$$

where erradd = 0.00

errmul = 1.00The linear absorption coefficient, μ, for Mo-Kα radiation is 21.121 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.704 to 0.810. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques[3]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[4] on F^2 was based on 2271 observed reflections and 104 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \; ||Fo| - |Fc|| \; / \; \Sigma \; |Fo| = 0.0344$$

$$wR2 = [\Sigma \; (\; w \; (Fo^2 - Fc^2)^2 \;) / \; \Sigma \; w(Fo^2)^2]^{1/2} = 0.0600$$

The standard deviation of an observation of unit weight[5] was 1.12. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.32 and -0.38 $e^-/Å^3$, respectively. Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All calculations were performed using the CrystalStructure[10] crystallographic software package except for refinement, which was performed using SHELXL-97[11].

References

(1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000.J.W.Pflugrath (1999) Acta Cryst. D55, 1718-1725.

(2) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(3) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(4) Least Squares function minimized: (SHELXL97)

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(5) Standard deviation of an observation of unit weight:

$$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$$

where: N_o = number of observations, N_v = number of variables

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(11) SHELX97: Sheldrick, G.M. (1997).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$IKCl_2O_6C_{12}H_{24}$
Formula Weight	501.23
Crystal Color, Habit	yellow, block
Crystal Dimensions	0.25 X 0.10 X 0.10 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	6 images @ 10.0 seconds
Detector Position	49.90 mm
Pixel Size	0.146 mm
Lattice Parameters	a = 8.8860(9) Å
	b = 8.1987(8) Å
	c = 13.9274(13) Å
	β = 101.873(2) ^{o}
	V = 992.96(16) $Å^3$
Space Group	$P2_1/n$ (#14)
Z value	2
D_{calc}	1.676 g/cm^3
F_{000}	500.00
μ(MoKα)	21.121 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku SCXmini
Radiation	MoKα (λ = 0.71075 Å)
	graphite monochromated
Detector Aperture	75 mm round
Data Images	540 exposures
ω oscillation Range (χ=54.0, ϕ=0.0)	-120.0 - 60.0^{o}
Exposure Rate	30.0 sec./^{o}
Detector Swing Angle	-28.40^{o}
ω oscillation Range (χ=54.0, ϕ=120.0)	-120.0 - 60.0^{o}
Exposure Rate	30.0 sec./^{o}
Detector Swing Angle	-28.40^{o}
ω oscillation Range (χ=54.0, ϕ=240.0)	-120.0 - 60.0^{o}
Exposure Rate	30.0 sec./^{o}
Detector Swing Angle	-28.40^{o}
Detector Position	49.90 mm
Pixel Size	0.146 mm
$2\theta_{max}$	55.0^{o}
No. of Reflections Measured	Total: 10037
	Unique: 2271 (R_{int} = 0.035)
Corrections	Lorentz-polarization
	Absorption
	(trans. factors: 0.704 - 0.810)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma\, w\, (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\, \sigma^2(Fo^2) + (0.0198 \cdot P)^2 + 0.4172 \cdot P\,]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	2271
No. Variables	104
Reflection/Parameter Ratio	21.84
Residuals: R1 (I>2.00σ(I))	0.0344
Residuals: R (All reflections)	0.0576
Residuals: wR2 (All reflections)	0.0600
Goodness of Fit Indicator	1.124
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.32 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.38 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
I(1)	1.0000	1.0000	1.0000	3.064(7)	1/2
K(1)	1.0000	0.5000	1.0000	3.42(5)	1/2
Cl(1)	0.78515(11)	0.81836(12)	1.03276(8)	5.53(2)	
O(1)	0.7229(2)	0.3920(2)	1.0415(2)	5.20(5)	
O(2)	0.7703(2)	0.3798(2)	0.85012(18)	5.21(5)	
O(3)	1.0219(3)	0.5552(2)	0.80380(18)	5.36(5)	
C(1)	0.6267(4)	0.2998(5)	0.9678(4)	7.45(13)	
C(2)	0.6208(4)	0.3824(5)	0.8733(4)	7.63(14)	
C(3)	0.7737(6)	0.4425(5)	0.7553(3)	7.92(15)	
C(4)	0.9341(7)	0.4371(5)	0.7414(2)	7.80(14)	
C(5)	1.1768(6)	0.5590(5)	0.7920(3)	7.56(13)	
C(6)	1.2633(5)	0.6753(5)	0.8634(4)	7.16(12)	

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
H(1)	0.5232	0.2921	0.9816	8.94	
H(2)	0.6677	0.1892	0.9657	8.94	
H(3)	0.5471	0.3268	0.8216	9.15	
H(4)	0.5867	0.4954	0.8772	9.15	
H(5)	0.7358	0.5550	0.7497	9.50	
H(6)	0.7075	0.3766	0.7049	9.50	
H(7)	0.9774	0.3282	0.7574	9.36	
H(8)	0.9369	0.4607	0.6728	9.36	
H(9)	1.1807	0.5930	0.7251	9.07	
H(10)	1.2224	0.4501	0.8031	9.07	
H(11)	1.3655	0.6941	0.8493	8.59	
H(12)	1.2089	0.7798	0.8592	8.59	

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
I(1)	0.03465(14)	0.04002(15)	0.03962(15)	0.00398(14)	0.00269(10)	-0.00022(15)
K(1)	0.03(5)	0.0608	0.0347	-0.0099	0.0070	-0.0015
Cl(1)	0.0522(5)	0.0628(5)	0.0987(7)	-0.0097(4)	0.0238(5)	0.0115(5)
O(1)	0.0490(14)	0.0474(14)	0.109(2)	-0.0093(11)	0.0355(14)	0.0015(14)
O(2)	0.0574(15)	0.0590(15)	0.0656(16)	0.0123(12)	-0.0250(12)	-0.0082(12)
O(3)	0.112(2)	0.0536(14)	0.0432(13)	0.0196(14)	0.0293(15)	0.0047(11)
C(1)	0.033(2)	0.057(2)	0.194(5)	-0.0113(18)	0.025(2)	-0.020(3)
C(2)	0.040(2)	0.069(3)	0.157(4)	0.013(2)	-0.035(2)	-0.041(3)
C(3)	0.152(5)	0.056(2)	0.061(2)	0.025(2)	-0.052(3)	-0.0117(19)
C(4)	0.195(6)	0.066(2)	0.034(2)	0.033(3)	0.019(3)	0.0005(19)
C(5)	0.156(4)	0.072(2)	0.088(3)	0.043(3)	0.093(3)	0.031(2)
C(6)	0.092(3)	0.058(2)	0.150(4)	0.015(2)	0.091(3)	0.033(3)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
I(1)	Cl(1)	2.5356(10)	I(1)	Cl(1)[1)]	2.5356(10)
K(1)	Cl(1)	3.3203(10)	K(1)	Cl(1)[2)]	3.3203(10)
K(1)	O(1)	2.787(2)	K(1)	O(1)[2)]	2.787(2)
K(1)	O(2)	2.782(2)	K(1)	O(2)[2)]	2.782(2)
K(1)	O(3)	2.816(2)	K(1)	O(3)[2)]	2.816(2)
O(1)	C(1)	1.413(5)	O(1)	C(6)[2)]	1.416(6)
O(2)	C(2)	1.430(5)	O(2)	C(3)	1.423(5)
O(3)	C(4)	1.423(5)	O(3)	C(5)	1.419(6)
C(1)	C(2)	1.471(8)	C(3)	C(4)	1.478(8)
C(5)	C(6)	1.474(6)			

Symmetry Operators:

(1) -X+2,-Y+2,-Z+2 (2) -X+2,-Y+1,-Z+2

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.980	C(1)	H(2)	0.980
C(2)	H(3)	0.980	C(2)	H(4)	0.980
C(3)	H(5)	0.980	C(3)	H(6)	0.980
C(4)	H(7)	0.980	C(4)	H(8)	0.980
C(5)	H(9)	0.980	C(5)	H(10)	0.980
C(6)	H(11)	0.980	C(6)	H(12)	0.980

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl(1)	I(1)	Cl(1)[1)]	180.00(4)	Cl(1)	K(1)	Cl(1)[2)]	180.00(3)
Cl(1)	K(1)	O(1)	70.36(5)	Cl(1)	K(1)	O(1)[2)]	109.64(5)
Cl(1)	K(1)	O(2)	91.59(5)	Cl(1)	K(1)	O(2)[2)]	88.41(5)
Cl(1)	K(1)	O(3)	99.41(5)	Cl(1)	K(1)	O(3)[2)]	80.59(5)
Cl(1)[2)]	K(1)	O(1)	109.64(5)	Cl(1)[2)]	K(1)	O(1)[2)]	70.36(5)
Cl(1)[2)]	K(1)	O(2)	88.41(5)	Cl(1)[2)]	K(1)	O(2)[2)]	91.59(5)
Cl(1)[2)]	K(1)	O(3)	80.59(5)	Cl(1)[2)]	K(1)	O(3)[2)]	99.41(5)
O(1)	K(1)	O(1)[2)]	0(440)	O(1)	K(1)	O(2)	60.01(7)
O(1)	K(1)	O(2)[2)]	119.99(7)	O(1)	K(1)	O(3)	119.54(8)
O(1)	K(1)	O(3)[2)]	60.46(8)	O(1)[2)]	K(1)	O(2)	119.99(7)
O(1)[2)]	K(1)	O(2)[2)]	60.01(7)	O(1)[2)]	K(1)	O(3)	60.46(8)
O(1)[2)]	K(1)	O(3)[2)]	119.54(8)	O(2)	K(1)	O(2)[2)]	180.00(10)
O(2)	K(1)	O(3)	61.02(7)	O(2)	K(1)	O(3)[2)]	118.98(7)
O(2)[2)]	K(1)	O(3)	118.98(7)	O(2)[2)]	K(1)	O(3)[2)]	61.02(7)
O(3)	K(1)	O(3)[2)]	180.00(10)	I(1)	Cl(1)	K(1)	87.79(2)
K(1)	O(1)	C(1)	116.6(2)	K(1)	O(1)	C(6)[2)]	114.0(2)
C(1)	O(1)	C(6)[2)]	113.3(3)	K(1)	O(2)	C(2)	113.4(2)
K(1)	O(2)	C(3)	114.6(2)	C(2)	O(2)	C(3)	113.7(3)
K(1)	O(3)	C(4)	110.1(2)	K(1)	O(3)	C(5)	112.2(2)
C(4)	O(3)	C(5)	111.6(3)	O(1)	C(1)	C(2)	108.0(3)
O(2)	C(2)	C(1)	109.3(3)	O(2)	C(3)	C(4)	108.4(3)
O(3)	C(4)	C(3)	108.8(3)	O(3)	C(5)	C(6)	108.6(4)

O(1)[2)]	C(6)	C(5)	108.2(3)

Symmetry Operators:

(1) -X+2,-Y+2,-Z+2 (2) -X+2,-Y+1,-Z+2

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	C(1)	H(1)	110.1	O(1)	C(1)	H(2)	110.1
C(2)	C(1)	H(1)	110.1	C(2)	C(1)	H(2)	110.1
H(1)	C(1)	H(2)	108.4	O(2)	C(2)	H(3)	109.8
O(2)	C(2)	H(4)	109.8	C(1)	C(2)	H(3)	109.8
C(1)	C(2)	H(4)	109.8	H(3)	C(2)	H(4)	108.2
O(2)	C(3)	H(5)	110.0	O(2)	C(3)	H(6)	110.0
C(4)	C(3)	H(5)	110.0	C(4)	C(3)	H(6)	110.0
H(5)	C(3)	H(6)	108.4	O(3)	C(4)	H(7)	109.9
O(3)	C(4)	H(8)	109.9	C(3)	C(4)	H(7)	109.9
C(3)	C(4)	H(8)	109.9	H(7)	C(4)	H(8)	108.3
O(3)	C(5)	H(9)	110.0	O(3)	C(5)	H(10)	110.0
C(6)	C(5)	H(9)	110.0	C(6)	C(5)	H(10)	110.0
H(9)	C(5)	H(10)	108.3	O(1)[1)]	C(6)	H(11)	110.1
O(1)[1)]	C(6)	H(12)	110.1	C(5)	C(6)	H(11)	110.1
C(5)	C(6)	H(12)	110.1	H(11)	C(6)	H(12)	108.4

Symmetry Operators:

(1) -X+2,-Y+1,-Z+2

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
Cl(1)	K(1)	O(1)	C(1)	117.7(2)	Cl(1)	K(1)	O(1)	C(6)[1)]	-107.3(2)
O(1)	K(1)	Cl(1)	I(1)	-179.80(6)	Cl(1)	K(1)	O(1)[1)]	C(1)[1)]	62.3(2)
Cl(1)	K(1)	O(1)[1)]	C(6)	-72.7(2)	O(1)[1)]	K(1)	Cl(1)	I(1)	0.20(6)
Cl(1)	K(1)	O(2)	C(2)	-45.7(2)	Cl(1)	K(1)	O(2)	C(3)	87.1(2)
O(2)	K(1)	Cl(1)	I(1)	-122.55(5)	Cl(1)	K(1)	O(2)[1)]	C(2)[1)]	-134.3(2)
Cl(1)	K(1)	O(2)[1)]	C(3)[1)]	92.9(2)	O(2)[1)]	K(1)	Cl(1)	I(1)	57.45(5)
Cl(1)	K(1)	O(3)	C(4)	-109.2(2)	Cl(1)	K(1)	O(3)	C(5)	125.9(2)
O(3)	K(1)	Cl(1)	I(1)	-61.67(6)	Cl(1)	K(1)	O(3)[1)]	C(4)[1)]	-70.8(2)
Cl(1)	K(1)	O(3)[1)]	C(5)[1)]	54.1(2)	O(3)[1)]	K(1)	Cl(1)	I(1)	118.33(6)
Cl(1)[1)]	K(1)	O(1)	C(1)	-62.3(2)	Cl(1)[1)]	K(1)	O(1)	C(6)[1)]	72.7(2)
Cl(1)[1)]	K(1)	O(1)[1)]	C(1)[1)]	-117.7(2)	Cl(1)[1)]	K(1)	O(1)[1)]	C(6)	107.3(2)
Cl(1)[1)]	K(1)	O(2)	C(2)	134.3(2)	Cl(1)[1)]	K(1)	O(2)	C(3)	-92.9(2)
Cl(1)[1)]	K(1)	O(2)[1)]	C(2)[1)]	45.7(2)	Cl(1)[1)]	K(1)	O(2)[1)]	C(3)[1)]	-87.1(2)
Cl(1)[1)]	K(1)	O(3)	C(4)	70.8(2)	Cl(1)[1)]	K(1)	O(3)	C(5)	-54.1(2)
Cl(1)[1)]	K(1)	O(3)[1)]	C(4)[1)]	109.2(2)	Cl(1)[1)]	K(1)	O(3)[1)]	C(5)[1)]	-125.9(2)
O(1)	K(1)	O(2)	C(2)	20.5(2)	O(1)	K(1)	O(2)	C(3)	153.3(2)
O(2)	K(1)	O(1)	C(1)	13.8(2)	O(2)	K(1)	O(1)	C(6)[1)]	148.8(2)
O(1)	K(1)	O(2)[1)]	C(2)[1)]	159.5(2)	O(1)	K(1)	O(2)[1)]	C(3)[1)]	26.7(2)
O(2)[1)]	K(1)	O(1)	C(1)	-166.2(2)	O(2)[1)]	K(1)	O(1)	C(6)[1)]	-31.2(2)
O(1)	K(1)	O(3)	C(4)	-36.5(3)	O(1)	K(1)	O(3)	C(5)	-161.4(2)

O(3)	K(1)	O(1)	C(1)	27.9(2)	O(3)	K(1)	O(1)	C(6)[1)]	162.8(2)
O(1)	K(1)	O(3)[1)]	C(4)[1)]	-143.5(3)	O(1)	K(1)	O(3)[1)]	C(5)[1)]	-18.6(2)
O(3)[1)]	K(1)	O(1)	C(1)	-152.1(2)	O(3)[1)]	K(1)	O(1)	C(6)[1)]	-17.2(2)
O(1)[1)]	K(1)	O(2)	C(2)	-159.5(2)	O(1)[1)]	K(1)	O(2)	C(3)	-26.7(2)
O(2)	K(1)	O(1)[1)]	C(1)[1)]	166.2(2)	O(2)	K(1)	O(1)[1)]	C(6)	31.2(2)
O(1)[1)]	K(1)	O(2)[1)]	C(2)[1)]	-20.5(2)	O(1)[1)]	K(1)	O(2)[1)]	C(3)[1)]	-153.3(2)
O(2)[1)]	K(1)	O(1)[1)]	C(1)[1)]	-13.8(2)	O(2)[1)]	K(1)	O(1)[1)]	C(6)	-148.8(2)
O(1)[1)]	K(1)	O(3)	C(4)	143.5(3)	O(1)[1)]	K(1)	O(3)	C(5)	18.6(2)
O(3)	K(1)	O(1)[1)]	C(1)[1)]	152.1(2)	O(3)	K(1)	O(1)[1)]	C(6)	17.2(2)
O(1)[1)]	K(1)	O(3)[1)]	C(4)[1)]	36.5(3)	O(1)[1)]	K(1)	O(3)[1)]	C(5)[1)]	161.4(2)
O(3)[1)]	K(1)	O(1)[1)]	C(1)[1)]	-27.9(2)	O(3)[1)]	K(1)	O(1)[1)]	C(6)	-162.8(2)
O(2)	K(1)	O(3)	C(4)	-22.6(2)	O(2)	K(1)	O(3)	C(5)	-147.5(2)
O(3)	K(1)	O(2)	C(2)	-145.6(2)	O(3)	K(1)	O(2)	C(3)	-12.7(2)
O(2)	K(1)	O(3)[1)]	C(4)[1)]	-157.4(2)	O(2)	K(1)	O(3)[1)]	C(5)[1)]	-32.5(2)
O(3)[1)]	K(1)	O(2)	C(2)	34.4(2)	O(3)[1)]	K(1)	O(2)	C(3)	167.3(2)
O(2)[1)]	K(1)	O(3)	C(4)	157.4(2)	O(2)[1)]	K(1)	O(3)	C(5)	32.5(2)
O(3)	K(1)	O(2)[1)]	C(2)[1)]	-34.4(2)	O(3)	K(1)	O(2)[1)]	C(3)[1)]	-167.3(2)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(2)[1)]	K(1)	O(3)[1)]	C(4)[1)]	22.6(2)	O(2)[1)]	K(1)	O(3)[1)]	C(5)[1)]	147.5(2)
O(3)[1)]	K(1)	O(2)[1)]	C(2)[1)]	145.6(2)	O(3)[1)]	K(1)	O(2)[1)]	C(3)[1)]	12.7(2)
K(1)	O(1)	C(1)	C(2)	-45.1(3)	K(1)	O(1)	C(6)[1)]	C(5)[1)]	50.4(4)
C(1)	O(1)	C(6)[1)]	C(5)[1)]	-173.1(3)	C(6)[1)]	O(1)	C(1)	C(2)	179.5(3)
K(1)	O(2)	C(2)	C(1)	-52.6(3)	K(1)	O(2)	C(3)	C(4)	45.8(3)
C(2)	O(2)	C(3)	C(4)	178.6(3)	C(3)	O(2)	C(2)	C(1)	174.1(3)
K(1)	O(3)	C(4)	C(3)	56.2(3)	K(1)	O(3)	C(5)	C(6)	-52.0(3)
C(4)	O(3)	C(5)	C(6)	-176.1(3)	C(5)	O(3)	C(4)	C(3)	-178.5(3)
O(1)	C(1)	C(2)	O(2)	64.8(4)	O(2)	C(3)	C(4)	O(3)	-69.8(4)
O(3)	C(5)	C(6)	O(1)[1)]	69.4(4)					

Symmetry Operators:

(1) -X+2,-Y+1,-Z+2

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
I(1)	H(2)[1)]	3.282	I(1)	H(6)[2)]	3.225
I(1)	H(6)[3)]	3.225	Cl(1)	H(1)[4)]	2.853
Cl(1)	H(2)[1)]	3.287	Cl(1)	H(6)[2)]	3.358
Cl(1)	H(8)[2)]	3.335	Cl(1)	H(9)[5)]	3.097

O(1)	H(1)[4)]	3.362	O(1)	H(4)[4)]	3.314
O(2)	H(5)[6)]	2.999	O(3)	H(3)[2)]	2.818
O(3)	H(6)[2)]	3.319	C(1)	H(4)[4)]	3.572
C(1)	H(8)[6)]	3.381	C(2)	H(5)[6)]	3.558
C(2)	H(8)[6)]	3.535	C(2)	H(10)[7)]	3.517
C(2)	H(11)[7)]	3.388	C(3)	H(5)[6)]	3.178
C(4)	H(2)[2)]	3.512	C(4)	H(3)[2)]	3.327
C(4)	H(5)[6)]	3.491	C(4)	H(11)[8)]	3.108
C(5)	H(3)[2)]	3.160	C(5)	H(11)[8)]	3.558
C(5)	H(12)[8)]	3.404	C(6)	H(3)[2)]	3.586
C(6)	H(4)[9)]	3.200	C(6)	H(7)[10)]	3.363
C(6)	H(10)[10)]	3.254	H(1)	Cl(1)[4)]	2.853
H(1)	O(1)[4)]	3.362	H(1)	H(1)[4)]	3.484
H(1)	H(4)[4)]	2.940	H(1)	H(8)[6)]	3.529
H(1)	H(8)[11)]	3.579	H(1)	H(9)[6)]	3.478
H(1)	H(10)[7)]	3.502	H(2)	I(1)[12)]	3.282
H(2)	Cl(1)[12)]	3.287	H(2)	C(4)[6)]	3.512
H(2)	H(5)[6)]	3.464	H(2)	H(8)[6)]	2.712
H(3)	O(3)[6)]	2.818	H(3)	C(4)[6)]	3.327
H(3)	C(5)[6)]	3.160	H(3)	C(6)[6)]	3.586
H(3)	H(5)[6)]	3.235	H(3)	H(8)[6)]	3.006
H(3)	H(9)[6)]	2.767	H(3)	H(10)[7)]	3.018
H(3)	H(11)[7)]	3.475	H(3)	H(12)[6)]	3.050
H(4)	O(1)[4)]	3.314	H(4)	C(1)[4)]	3.572
H(4)	C(6)[7)]	3.200	H(4)	H(1)[4)]	2.940

H(4)	H(7)[2)]	3.293	H(4)	H(10)[7)]	3.211
H(4)	H(11)[7)]	2.521	H(5)	O(2)[2)]	2.999
H(5)	C(2)[2)]	3.558	H(5)	C(3)[2)]	3.178
H(5)	C(4)[2)]	3.491	H(5)	H(2)[2)]	3.464
H(5)	H(3)[2)]	3.235	H(5)	H(6)[2)]	2.734
H(5)	H(7)[2)]	2.921	H(6)	I(1)[6)]	3.225
H(6)	Cl(1)[6)]	3.358	H(6)	O(3)[6)]	3.319
H(6)	H(5)[6)]	2.734	H(7)	C(6)[8)]	3.363

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(7)	H(4)[6)]	3.293	H(7)	H(5)[6)]	2.921
H(7)	H(9)[8)]	3.565	H(7)	H(11)[8)]	2.494
H(7)	H(12)[8)]	3.526	H(8)	Cl(1)[6)]	3.335
H(8)	C(1)[2)]	3.381	H(8)	C(2)[2)]	3.535
H(8)	H(1)[2)]	3.529	H(8)	H(1)[13)]	3.579
H(8)	H(2)[2)]	2.712	H(8)	H(3)[2)]	3.006
H(8)	H(11)[8)]	2.860	H(8)	H(12)[8)]	3.589
H(9)	Cl(1)[14)]	3.097	H(9)	H(1)[2)]	3.478
H(9)	H(3)[2)]	2.767	H(9)	H(7)[10)]	3.565
H(9)	H(10)[10)]	3.099	H(9)	H(11)[8)]	3.429
H(9)	H(12)[8)]	3.067	H(10)	C(2)[9)]	3.517
H(10)	C(6)[8)]	3.254	H(10)	H(1)[9)]	3.502
H(10)	H(3)[9)]	3.018	H(10)	H(4)[9)]	3.211
H(10)	H(9)[8)]	3.099	H(10)	H(11)[8)]	2.973
H(10)	H(12)[8)]	2.828	H(11)	C(2)[9)]	3.388

H(11)	C(4)[10)]	3.108	H(11)	C(5)[10)]	3.558
H(11)	H(3)[9)]	3.475	H(11)	H(4)[9)]	2.521
H(11)	H(7)[10)]	2.494	H(11)	H(8)[10)]	2.860
H(11)	H(9)[10)]	3.429	H(11)	H(10)[10)]	2.973
H(12)	C(5)[10)]	3.404	H(12)	H(3)[2)]	3.050
H(12)	H(7)[10)]	3.526	H(12)	H(8)[10)]	3.589
H(12)	H(9)[10)]	3.067	H(12)	H(10)[10)]	2.828

Symmetry Operators:

(1) X,Y+1,Z (2) -X+1/2+1,Y+1/2,-Z+1/2+1

(3) X+1/2,-Y+1/2+1,Z+1/2 (4) -X+1,-Y+1,-Z+2

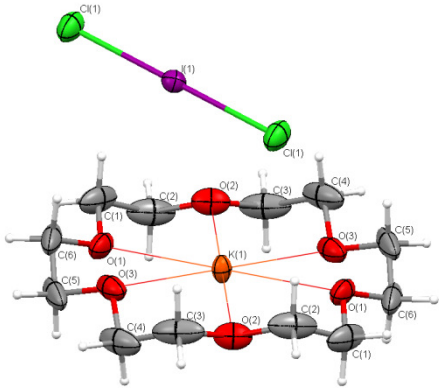
(5) X+1/2-1,-Y+1/2+1,Z+1/2 (6) -X+1/2+1,Y+1/2-1,-Z+1/2+1

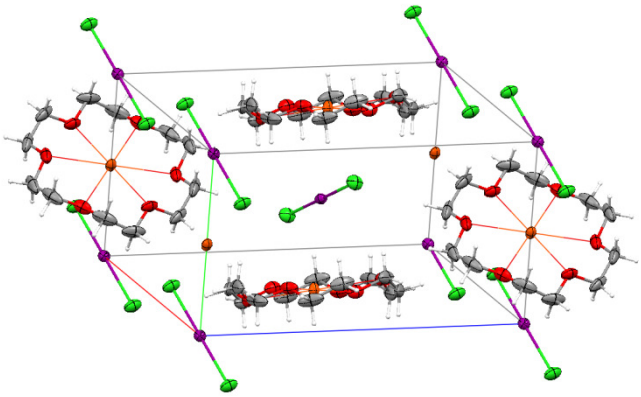
(7) X-1,Y,Z (8) -X+1/2+2,Y+1/2-1,-Z+1/2+1

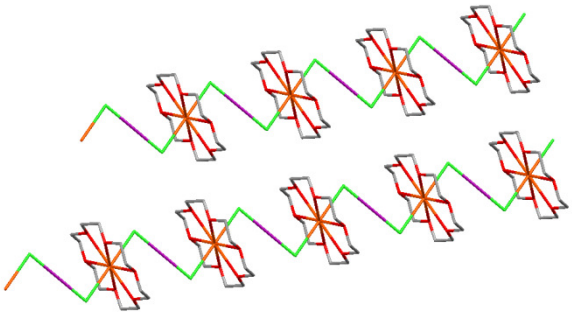
(9) X+1,Y,Z (10) -X+1/2+2,Y+1/2,-Z+1/2+1

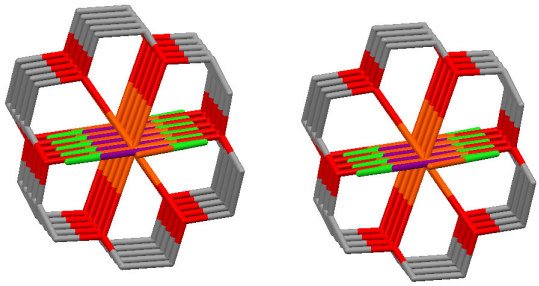
(11) X+1/2-1,-Y+1/2,Z+1/2 (12) X,Y-1,Z

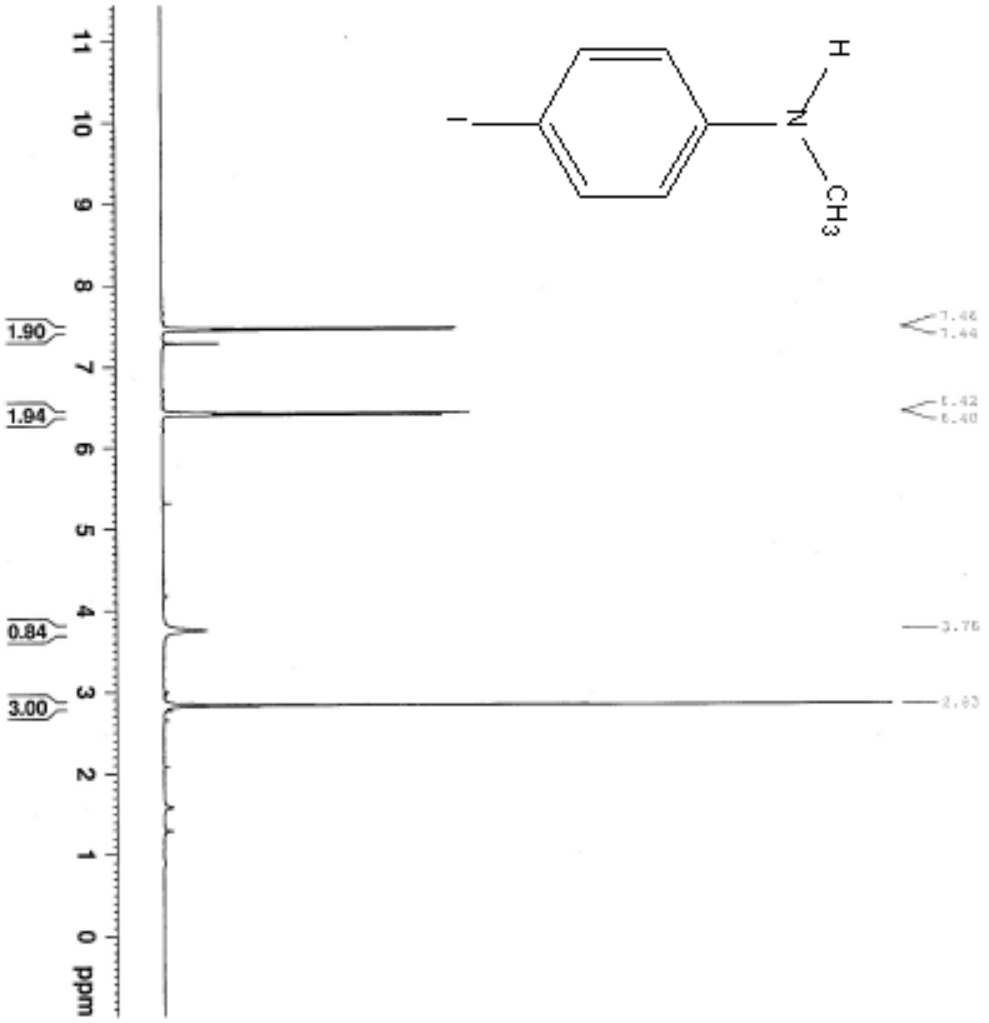
(13) X+1/2,-Y+1/2,Z+1/2-1 (14) X+1/2,-Y+1/2+1,Z+1/2-1

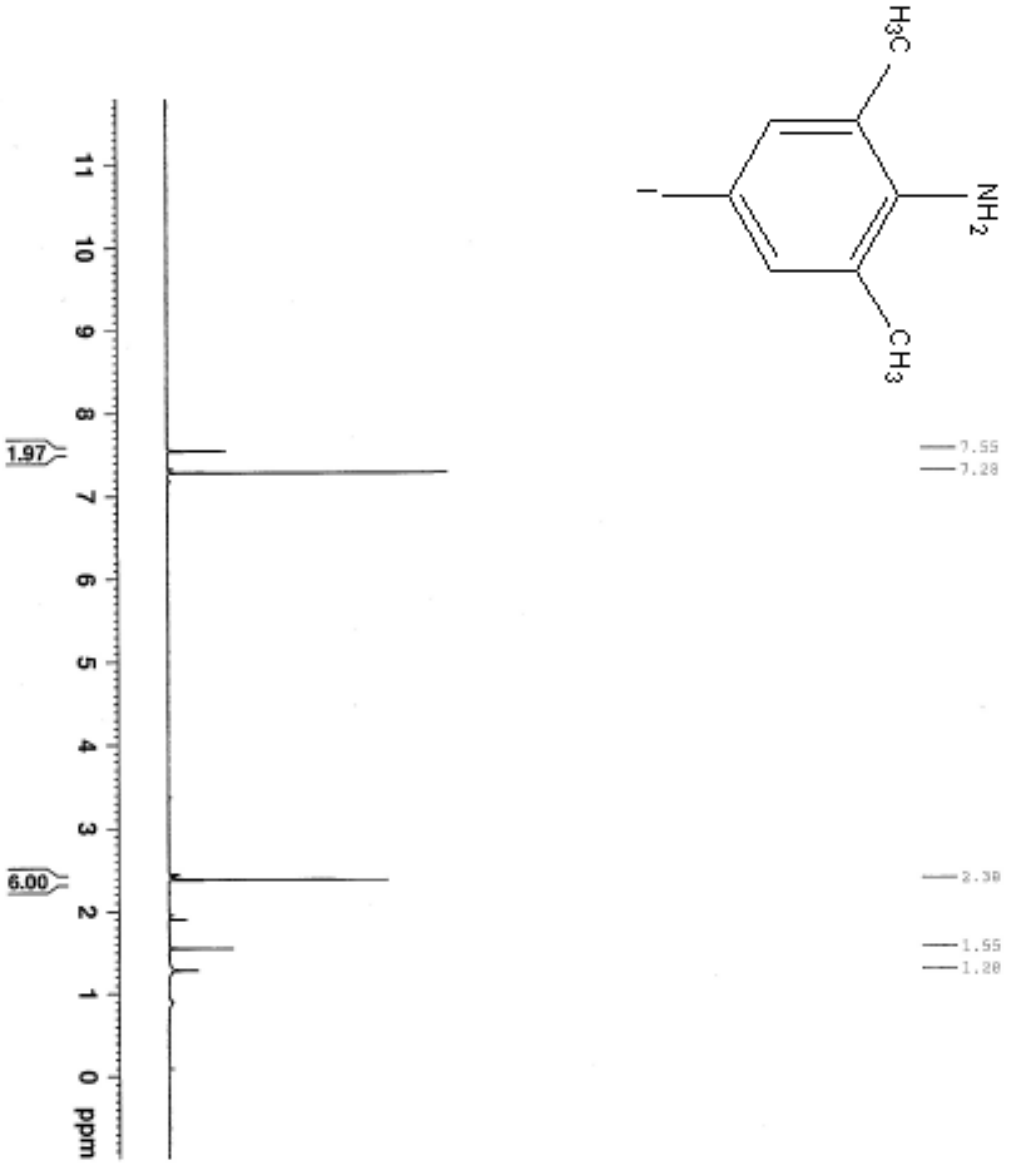

Cl(1)
K(1)
Cl(1)
O(2)
C(2)
C(3)
C(4)
C(1)
O(3)
C(5)
C(6)
O(1)
K(1)
O(3)
C(5)
O(1)
C(6)
C(4)
C(3)
O(2)
C(2)
C(1)

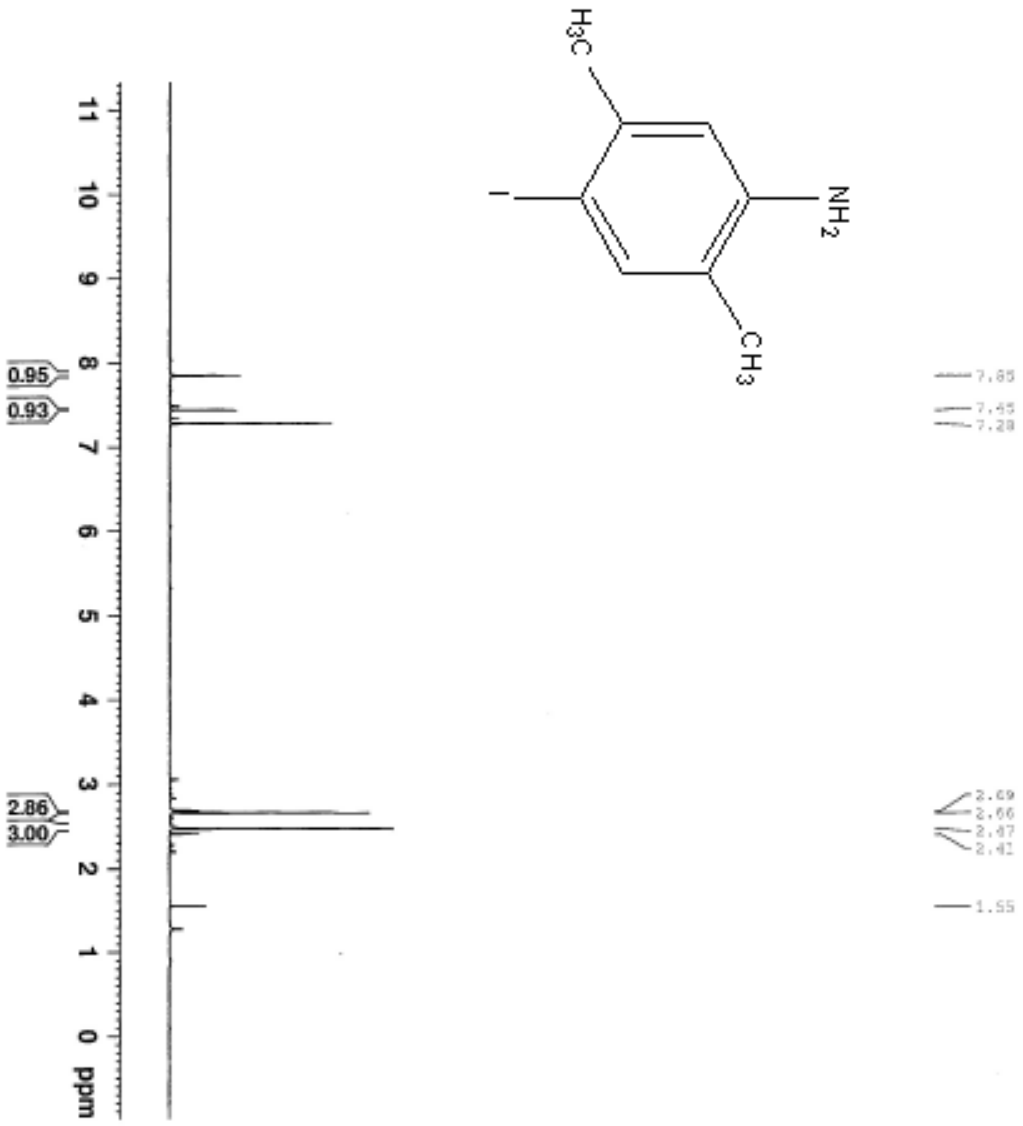


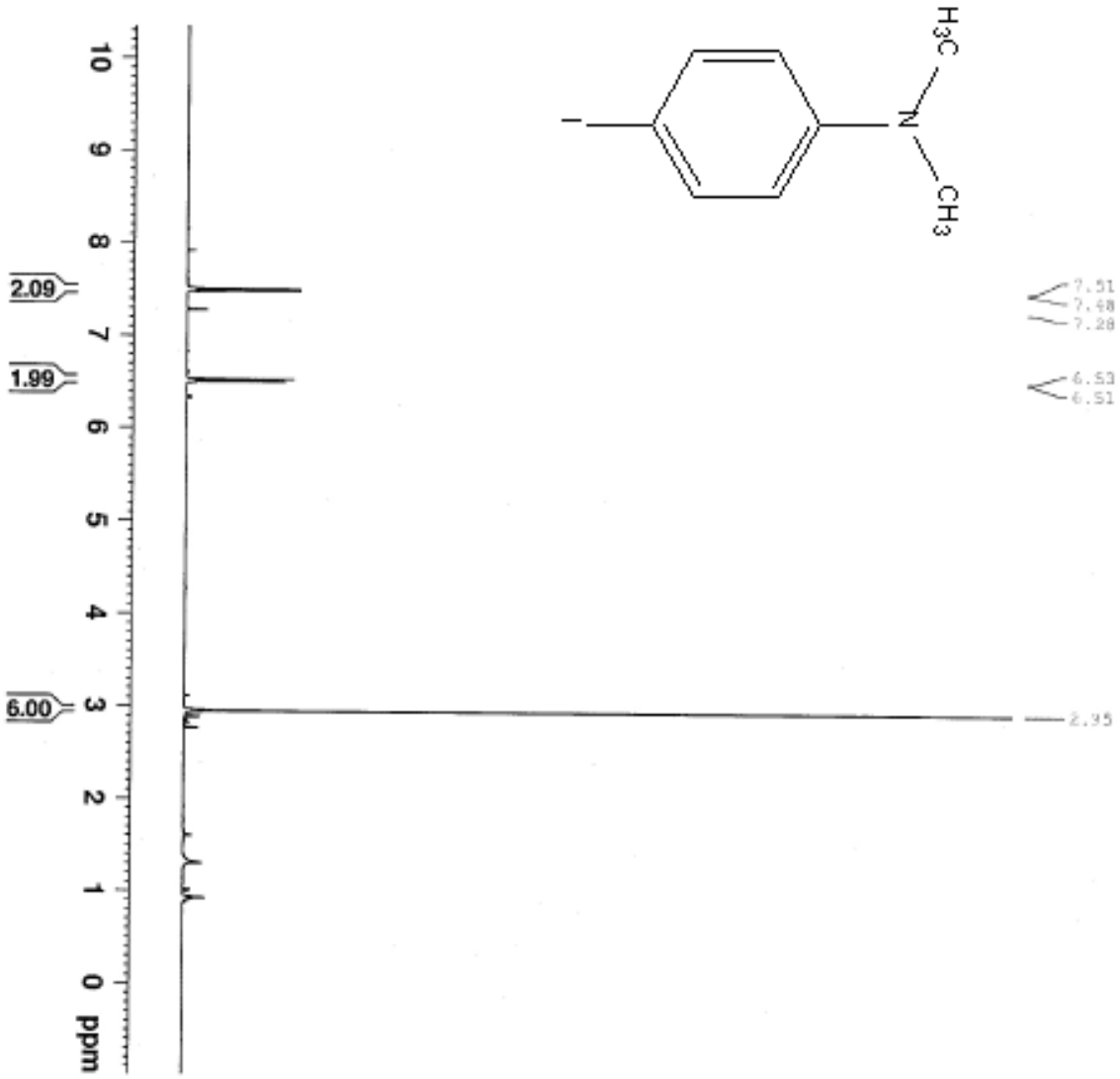


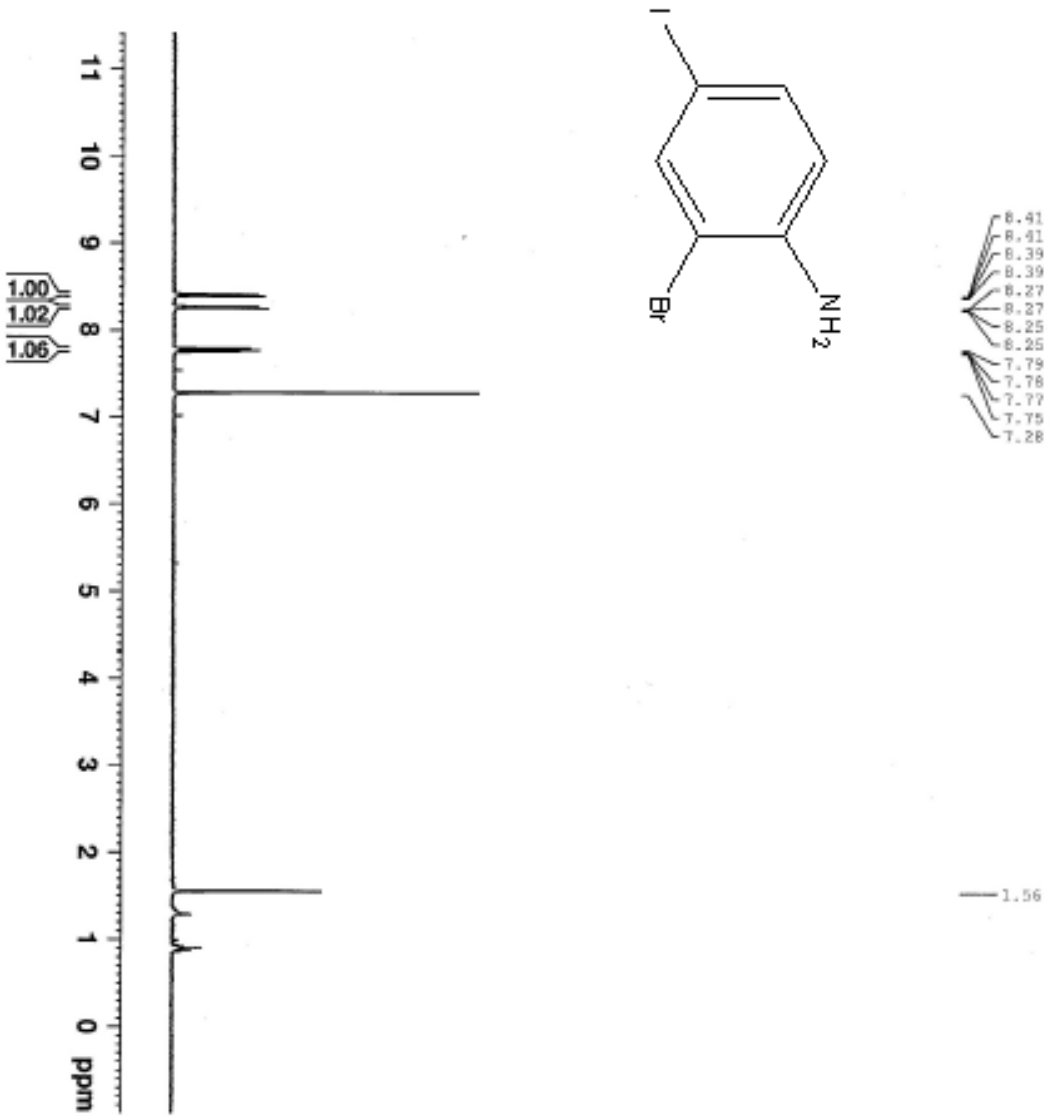


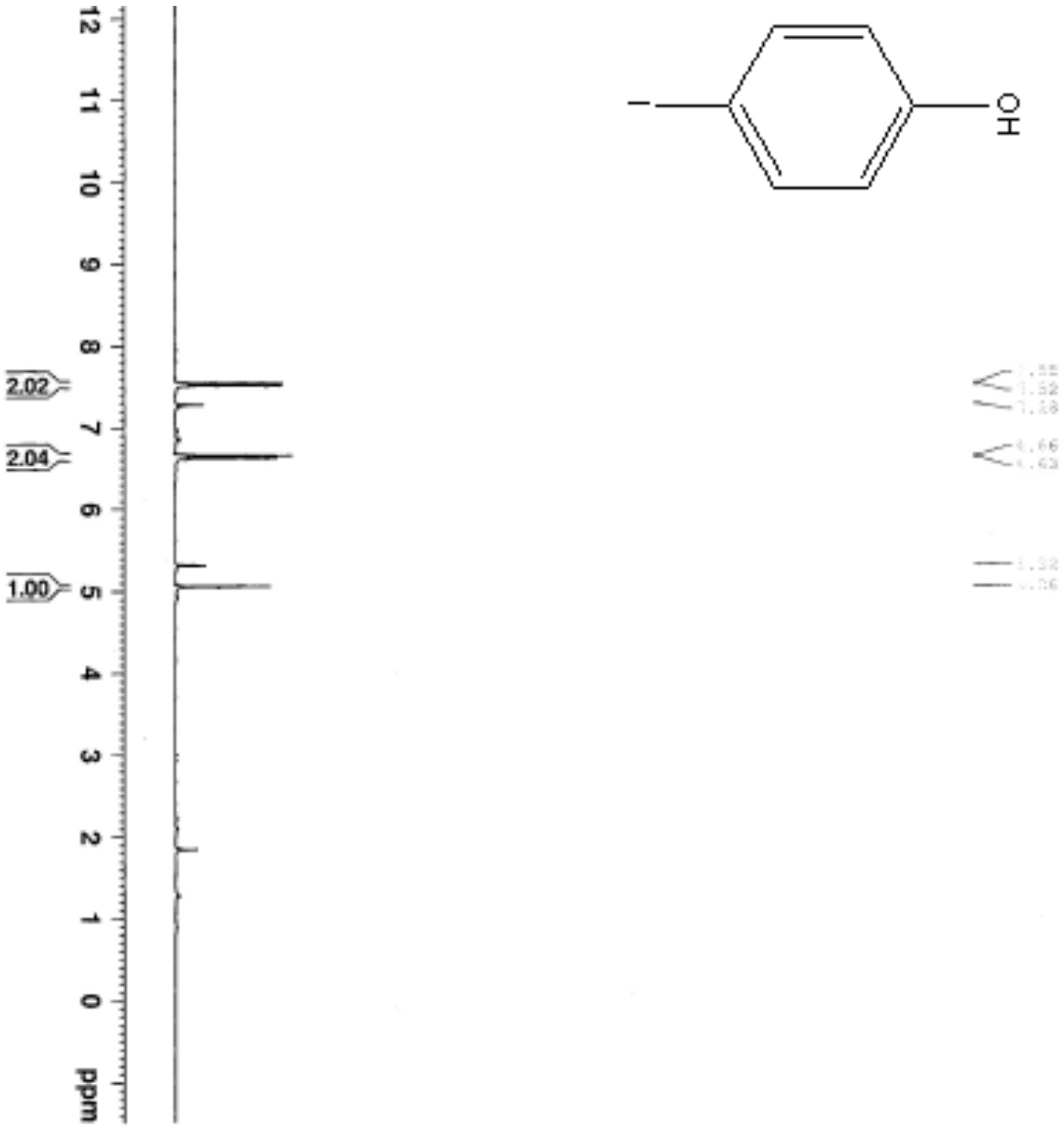

H
N
CH3
1.90
1.94
0.84
3.00
7.46
7.44
6.42
6.40
3.76
2.83
11
10
9
8
7
6
5
4
3
2
1
0
ppm

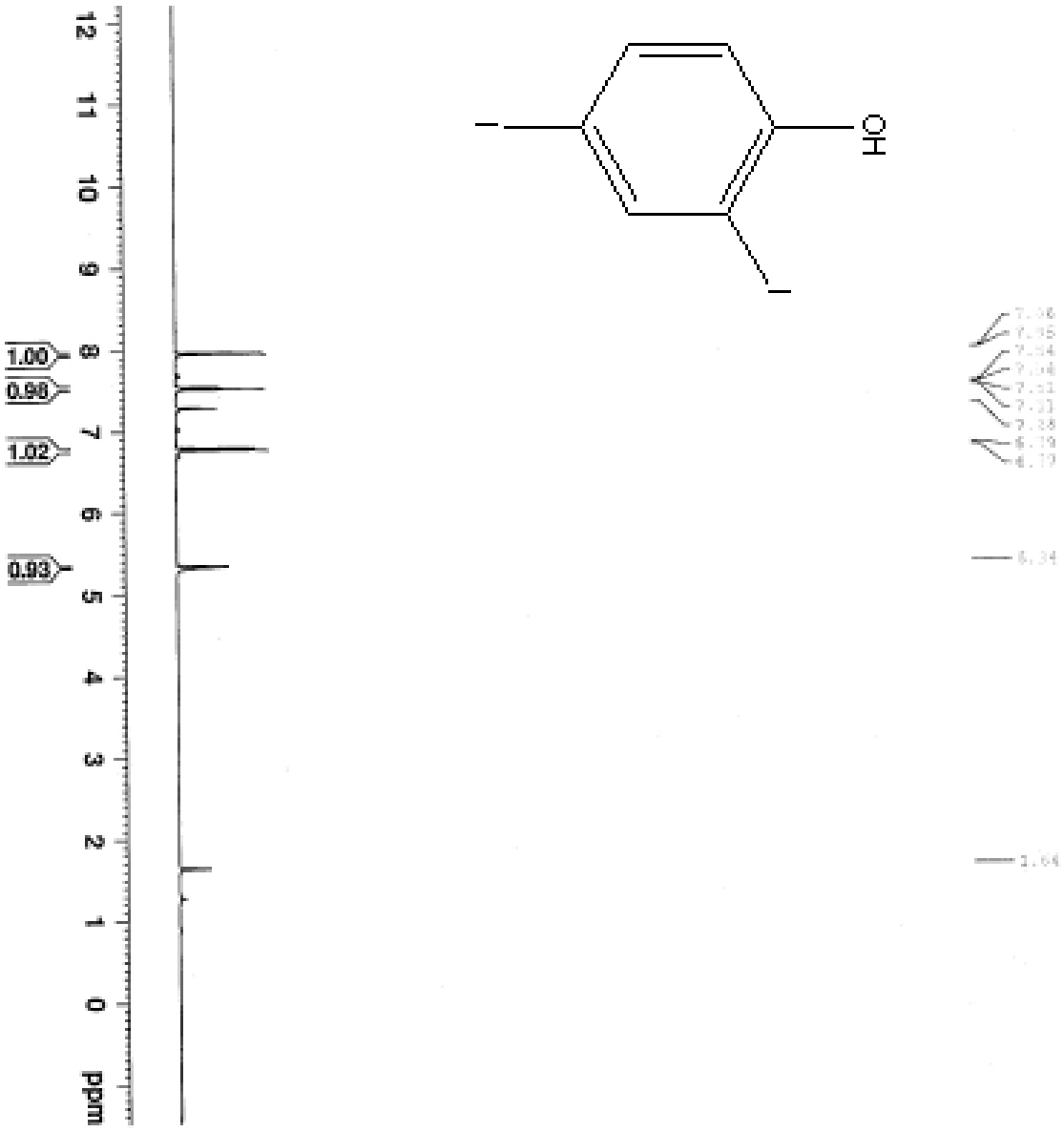

H3C
NH2
I
CH3
7.55
7.28
2.39
1.55
1.20
1.97
6.00
11
10
9
8
7
6
5
4
3
2
1
0
ppm

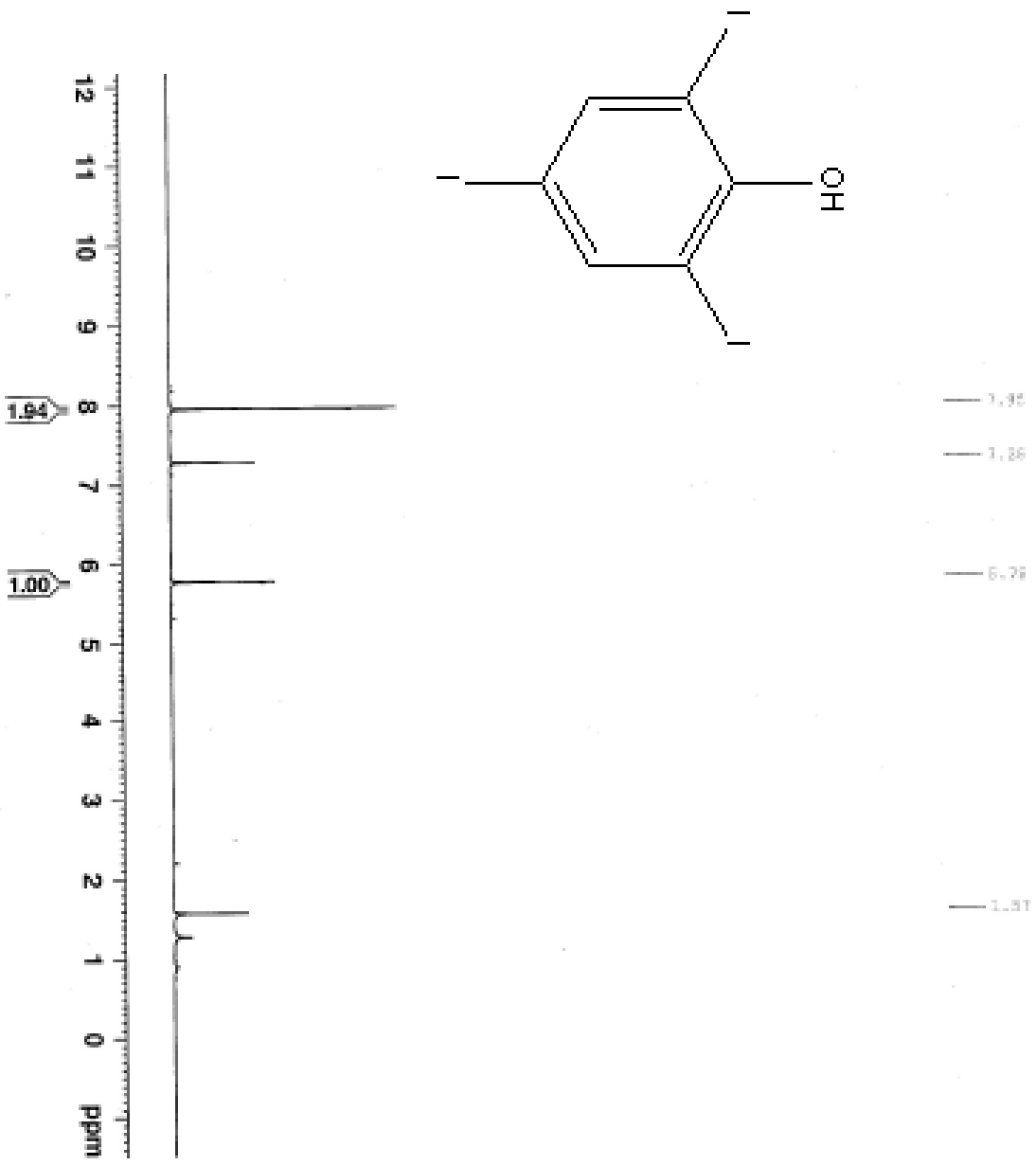

H3C
I
NH2
CH3
0.95
0.93
2.86
3.00
11
10
9
8
7
6
5
4
3
2
1
0
ppm
7.85
7.45
7.28
2.69
2.66
2.47
2.41
1.55

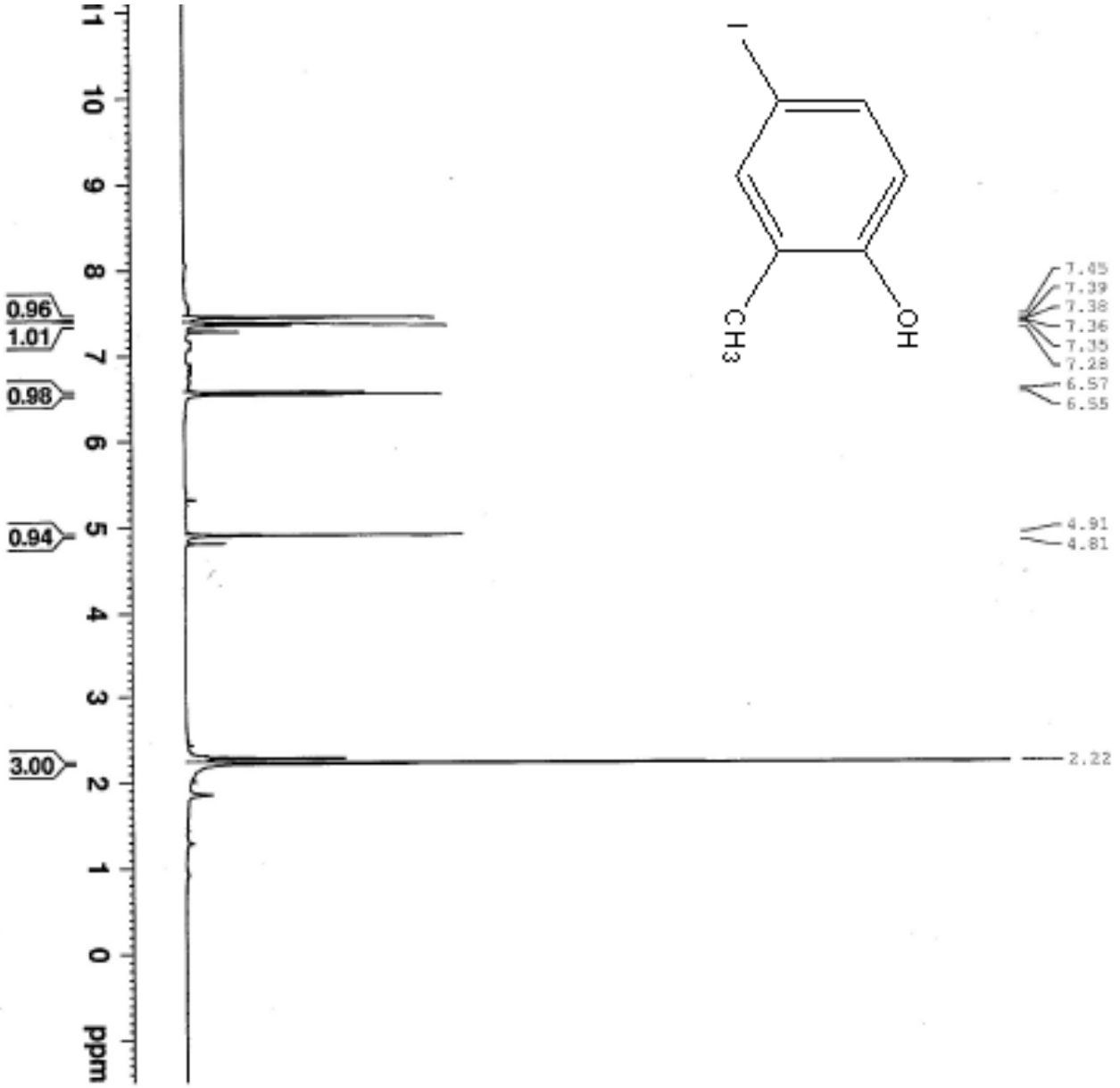

H3C
N
CH3
I
2.09
1.99
6.00
7.51
7.48
7.28
6.53
6.51
2.95
10
9
8
7
6
5
4
3
2
1
0
ppm

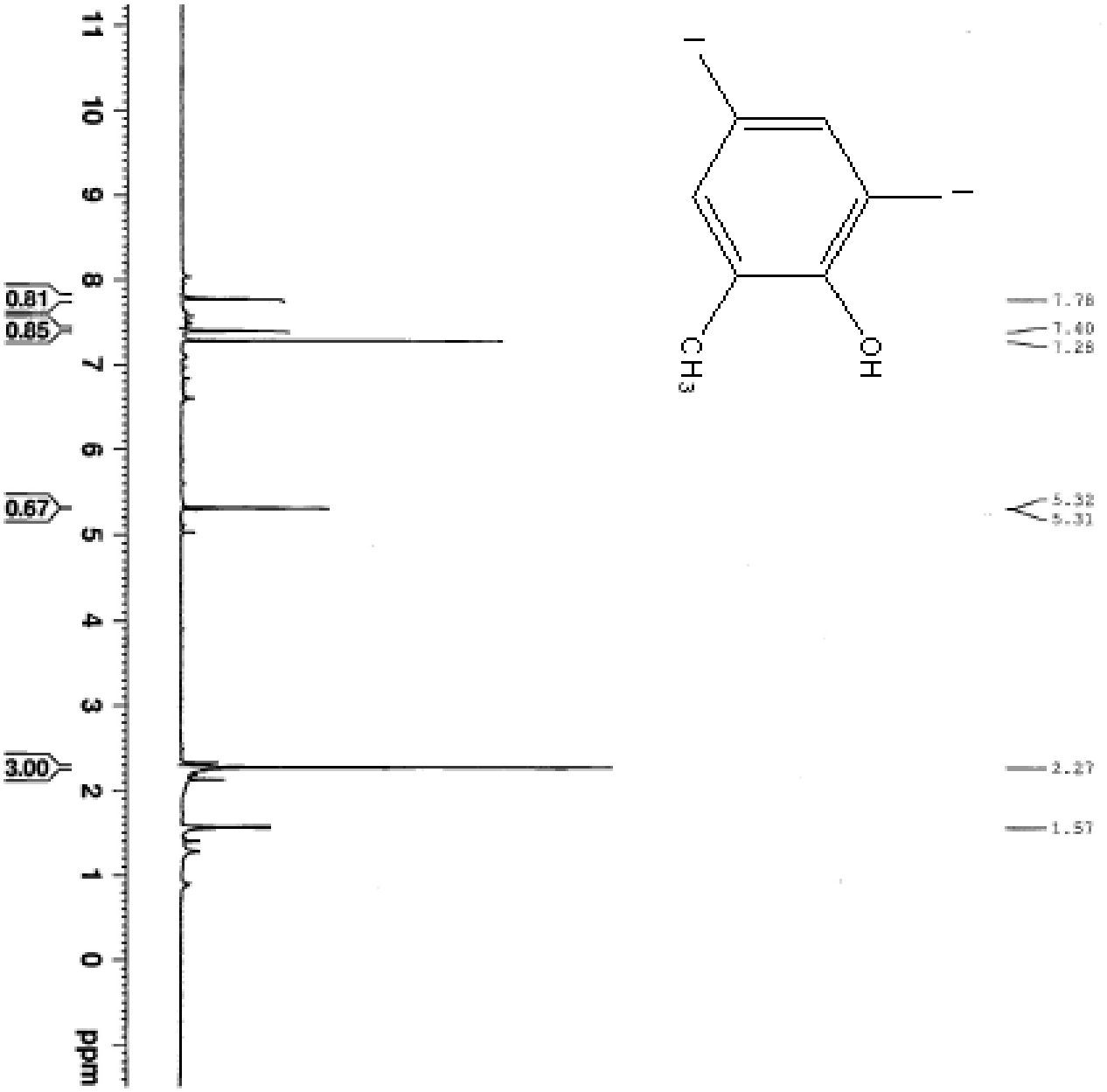

1.00
1.02
1.06
11
10
9
8
7
6
5
4
3
2
1
0
ppm
I
Br
NH2
8.41
8.41
8.39
8.39
8.27
8.27
8.25
8.25
7.79
7.78
7.77
7.75
7.28
1.56

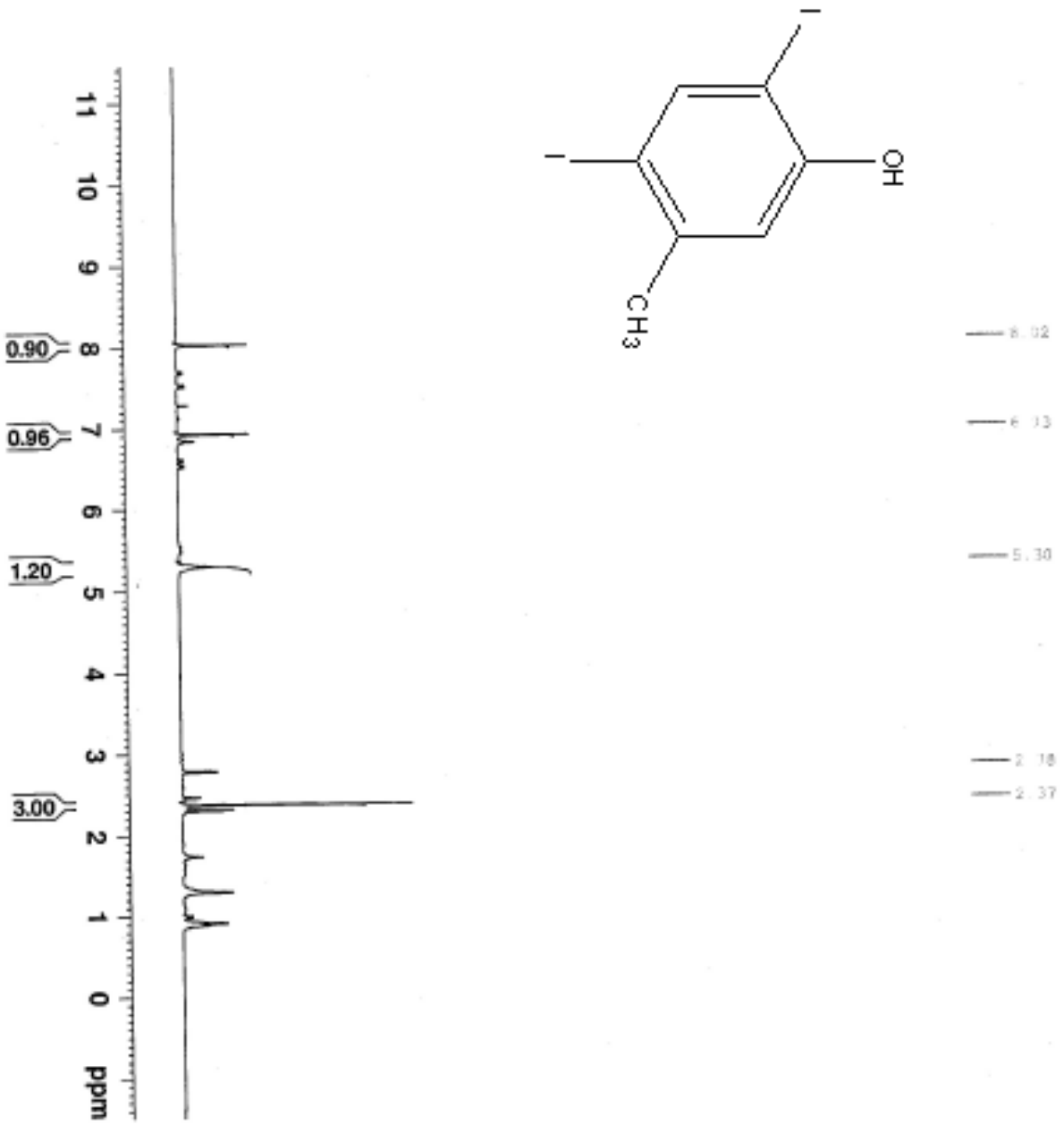

12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.02
2.04
1.00
OH

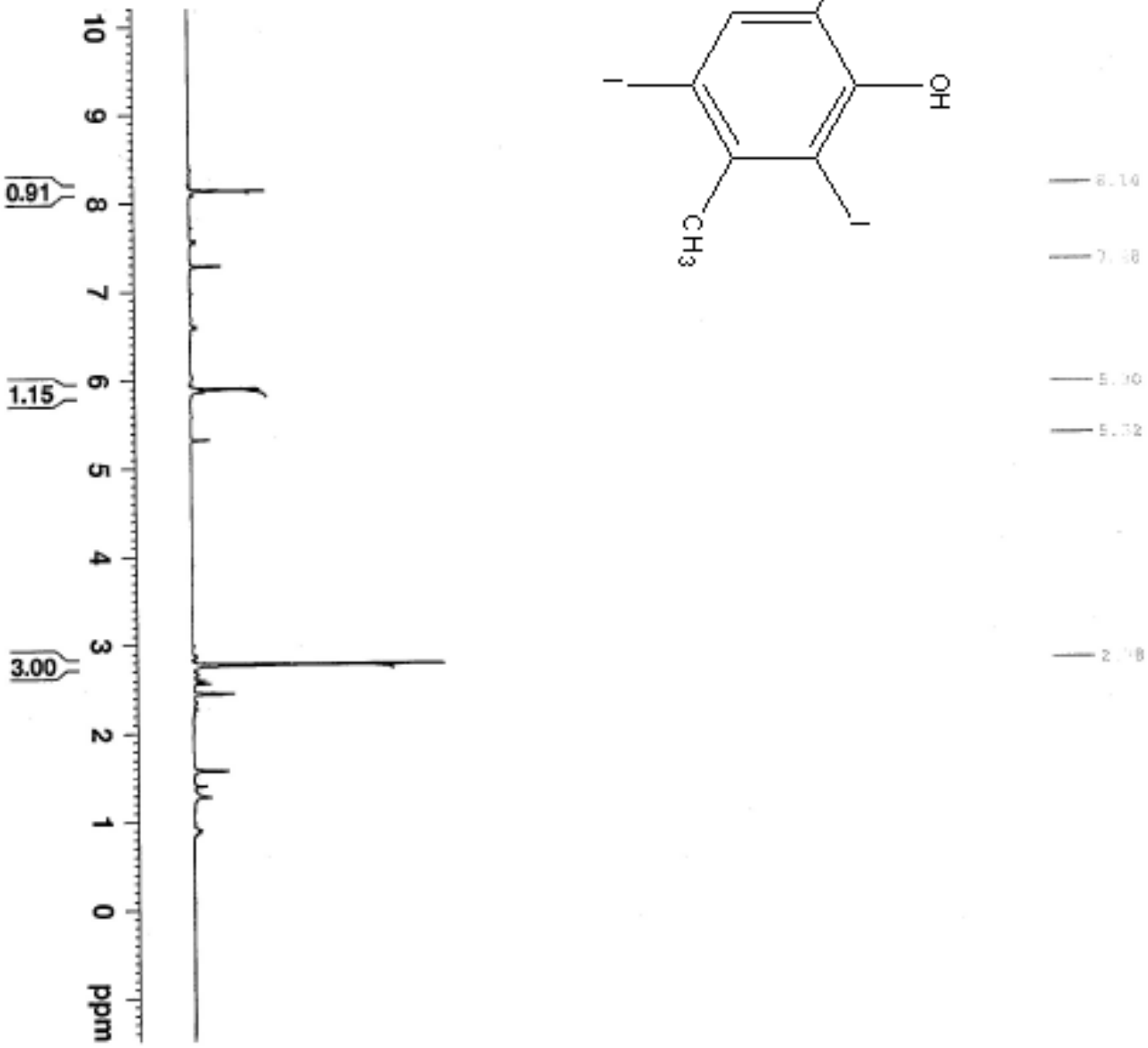

1.00
0.98
1.02
0.93
ppm
OH

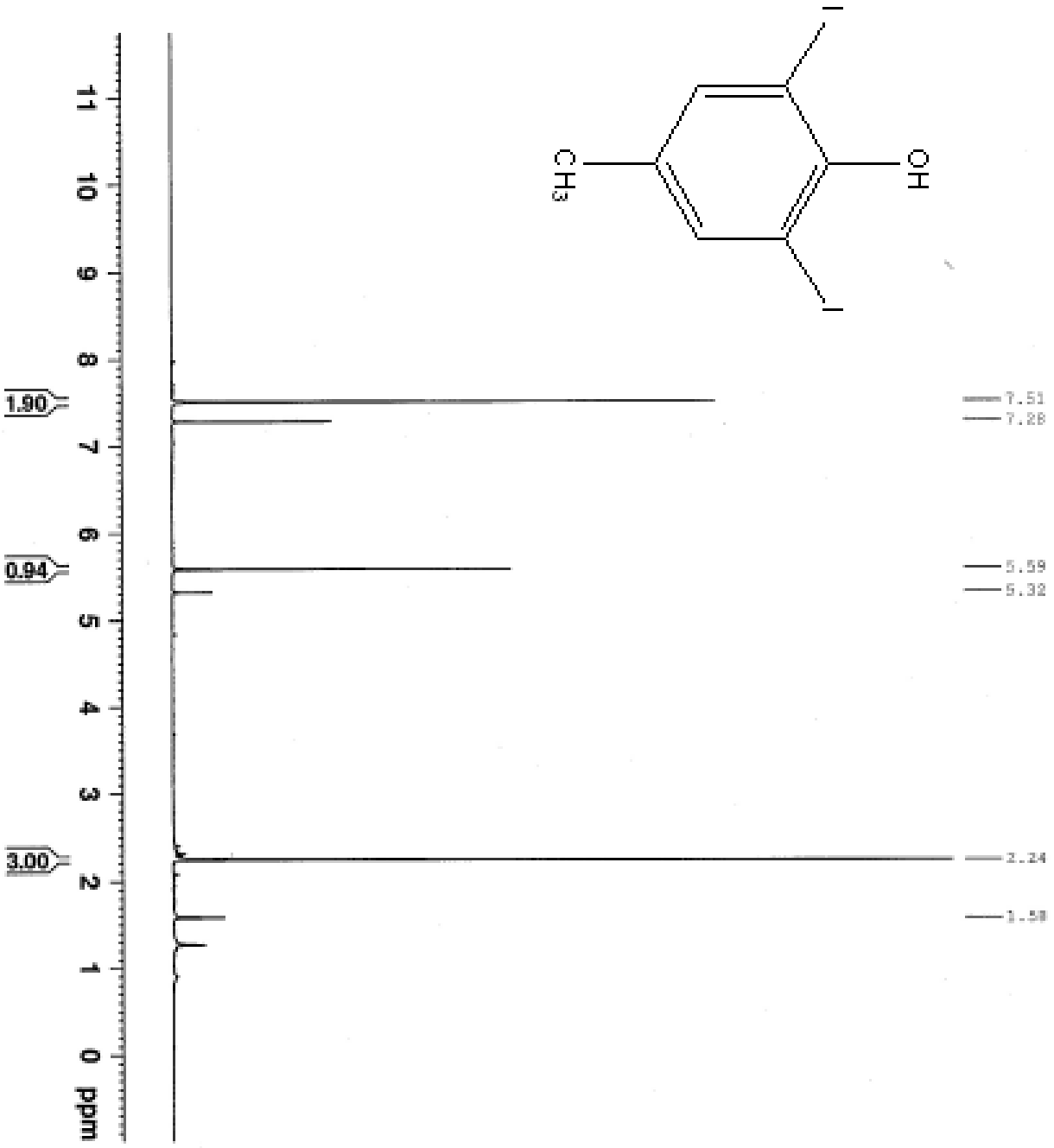

HO
1.94
1.00
ppm

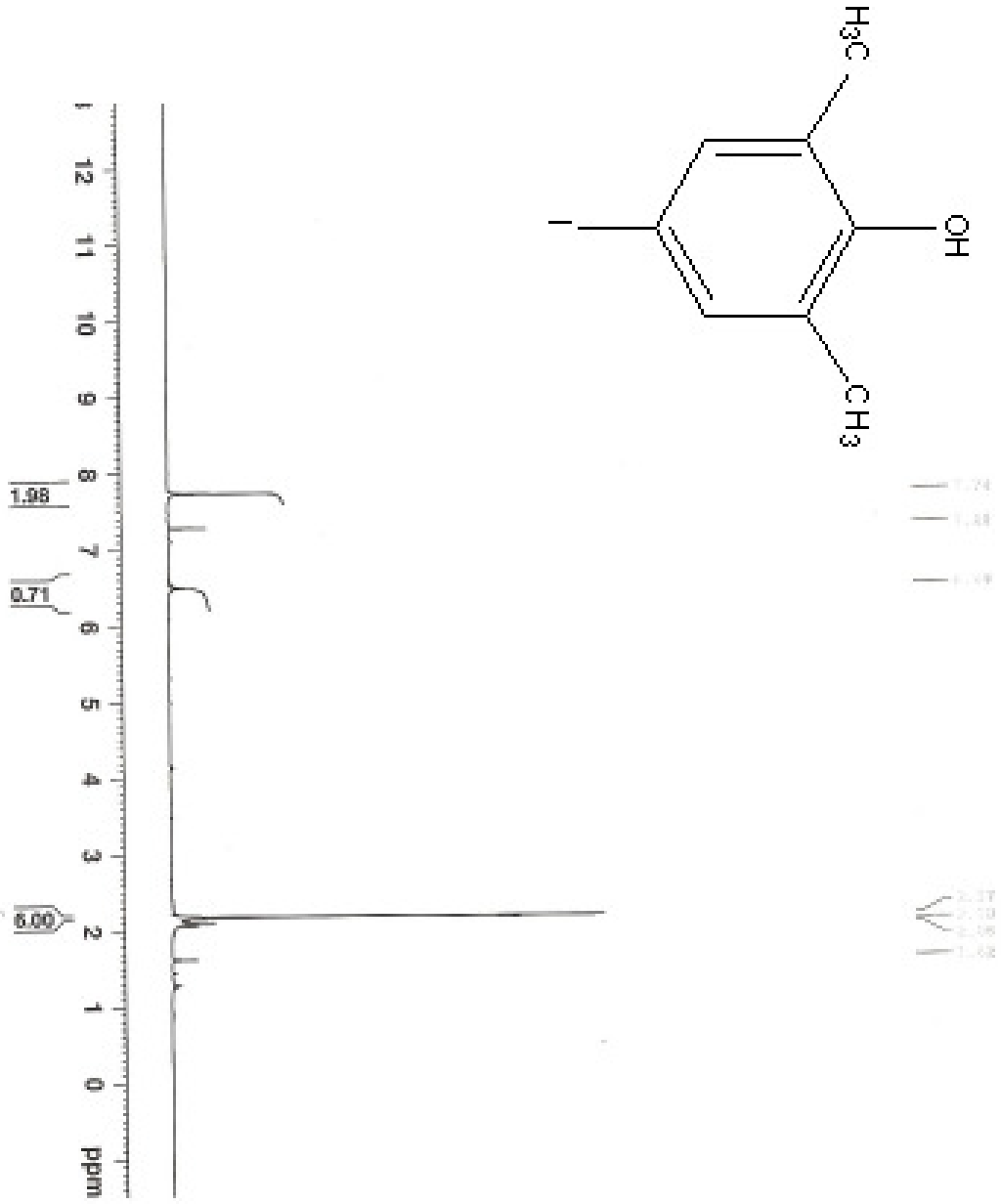

I
CH3
OH
7.45
7.39
7.38
7.36
7.35
7.28
6.57
6.55
4.91
4.81
2.22
0.96
1.01
0.98
0.94
3.00
11
10
9
8
7
6
5
4
3
2
1
0
ppm

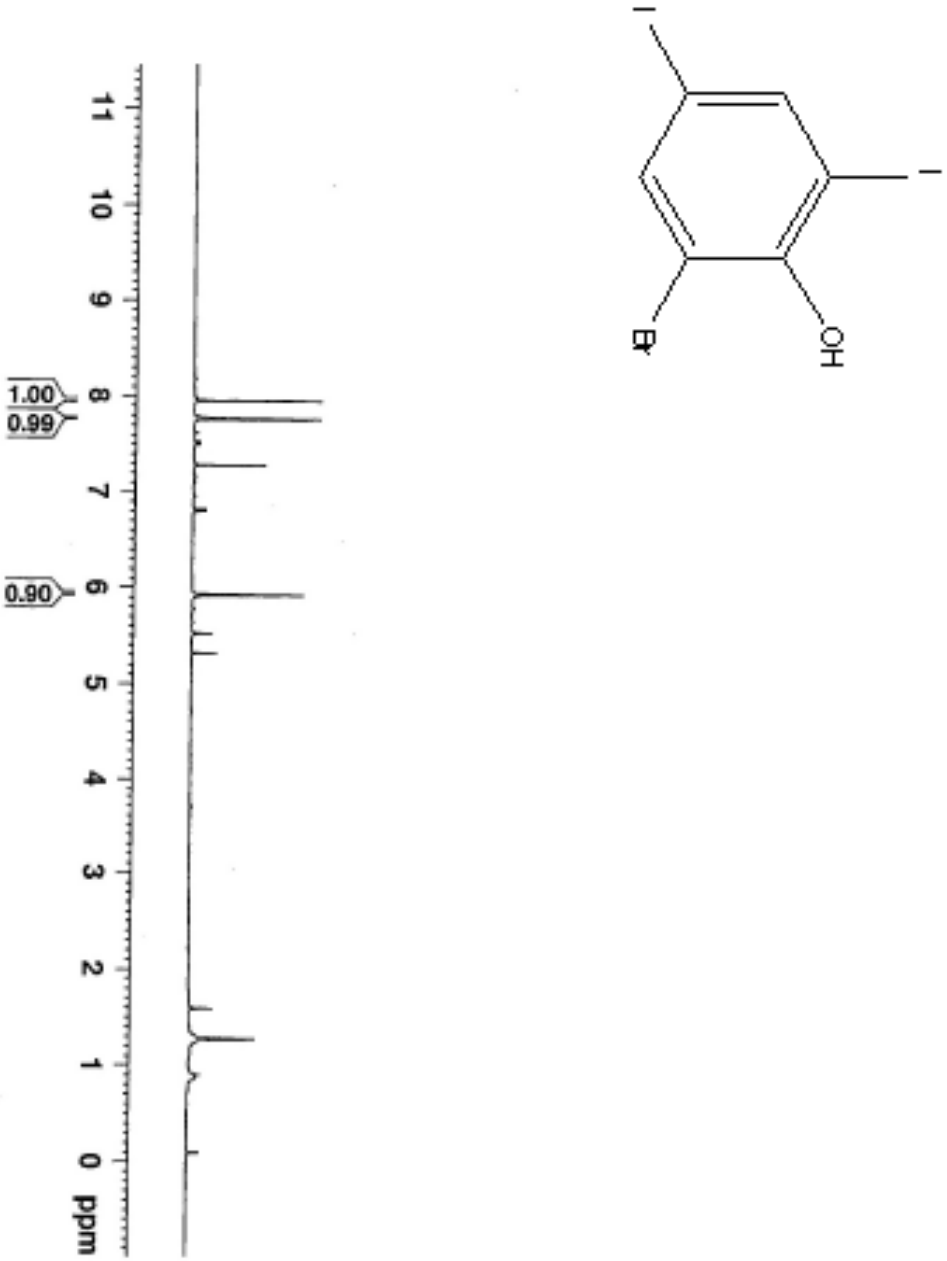

0.81
0.85
0.67
3.00
ppm
I
I
CH3
OH
7.76
7.40
7.28
5.32
5.31
2.27
1.57

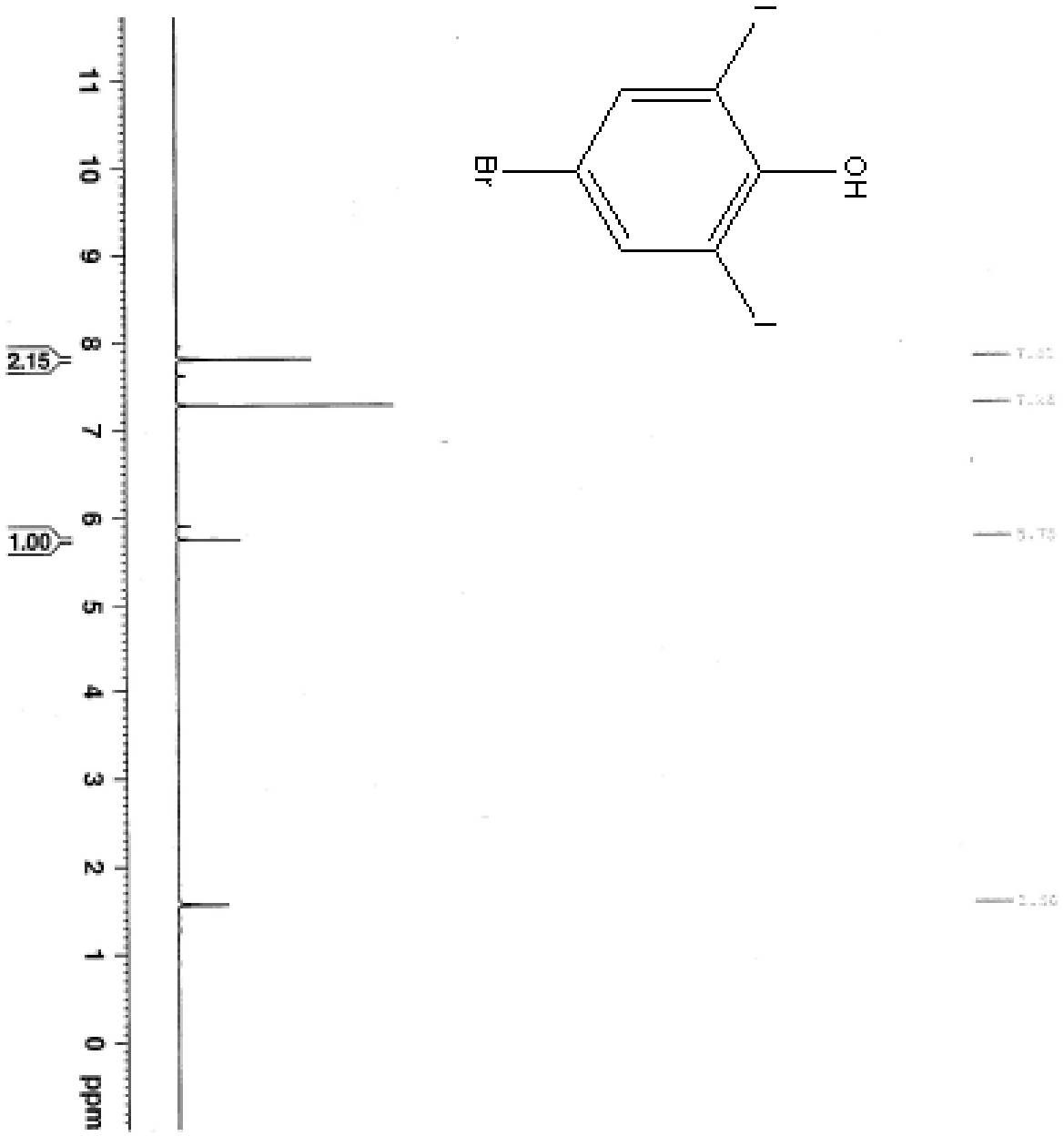

I
I
OH
CH3
8.02
6.13
5.30
2.18
2.37
0.90
0.96
1.20
3.00
11
10
9
8
7
6
5
4
3
2
1
0
ppm


0.91
1.15
3.00
10
9
8
7
6
5
4
3
2
1
0
ppm
I
I
I
HO
CH3
8.10
7.68
5.80
5.72
2.08

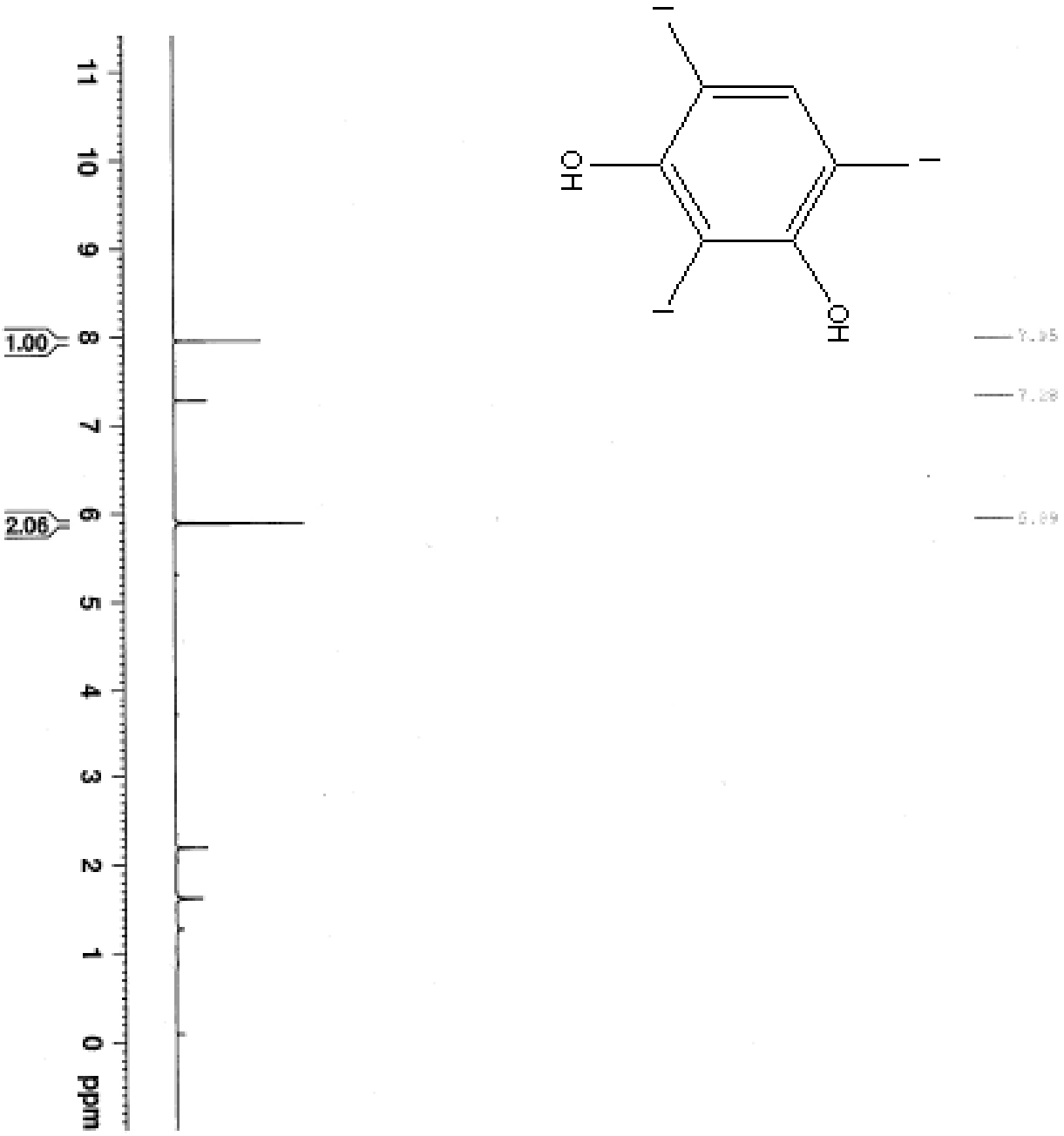

CH3
OH
1.90
0.94
3.00
7.51
7.28
5.59
5.32
2.24
1.58
ppm

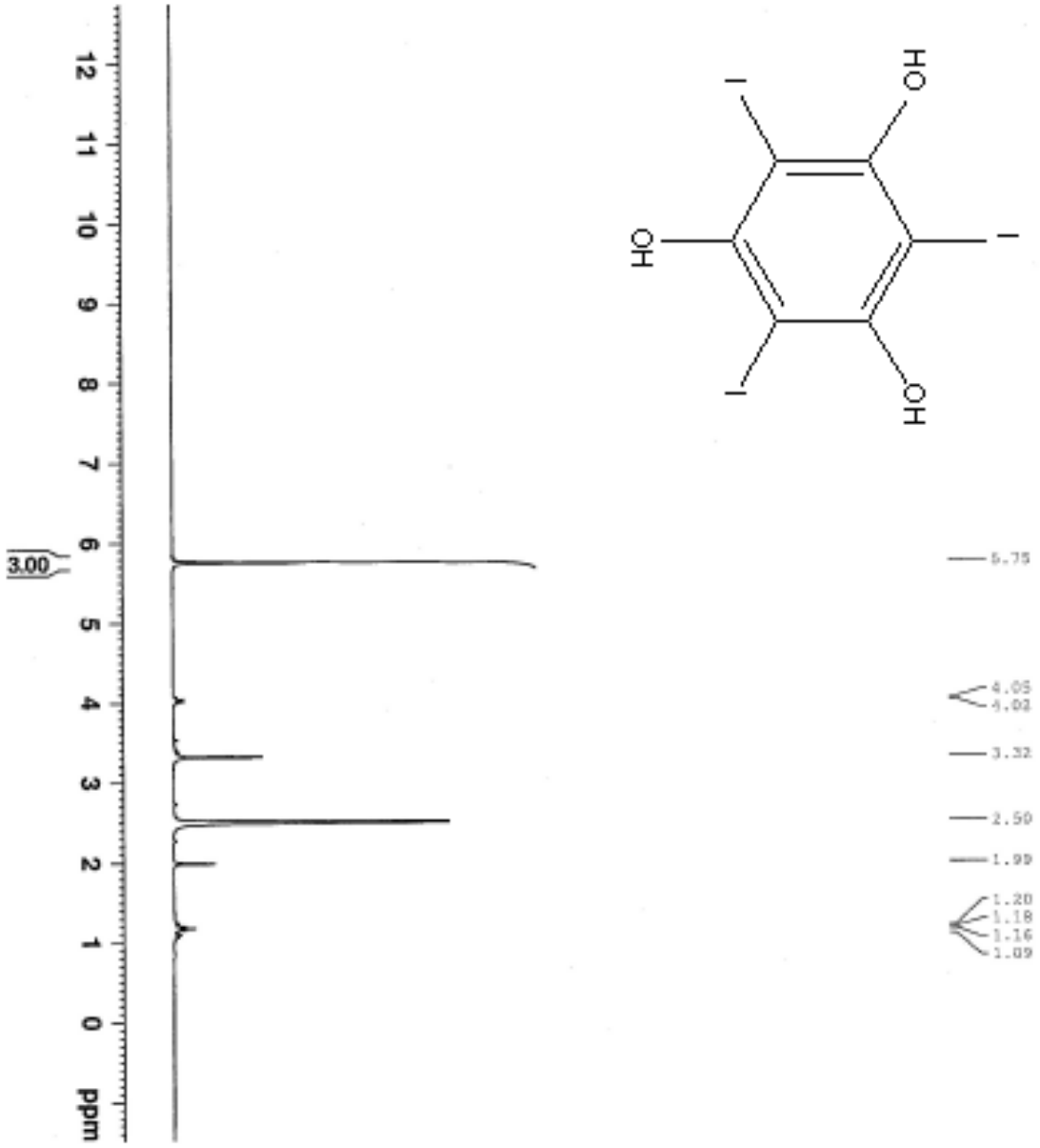

H3C
I
HO
CH3
1.98
0.71
6.00
ppm

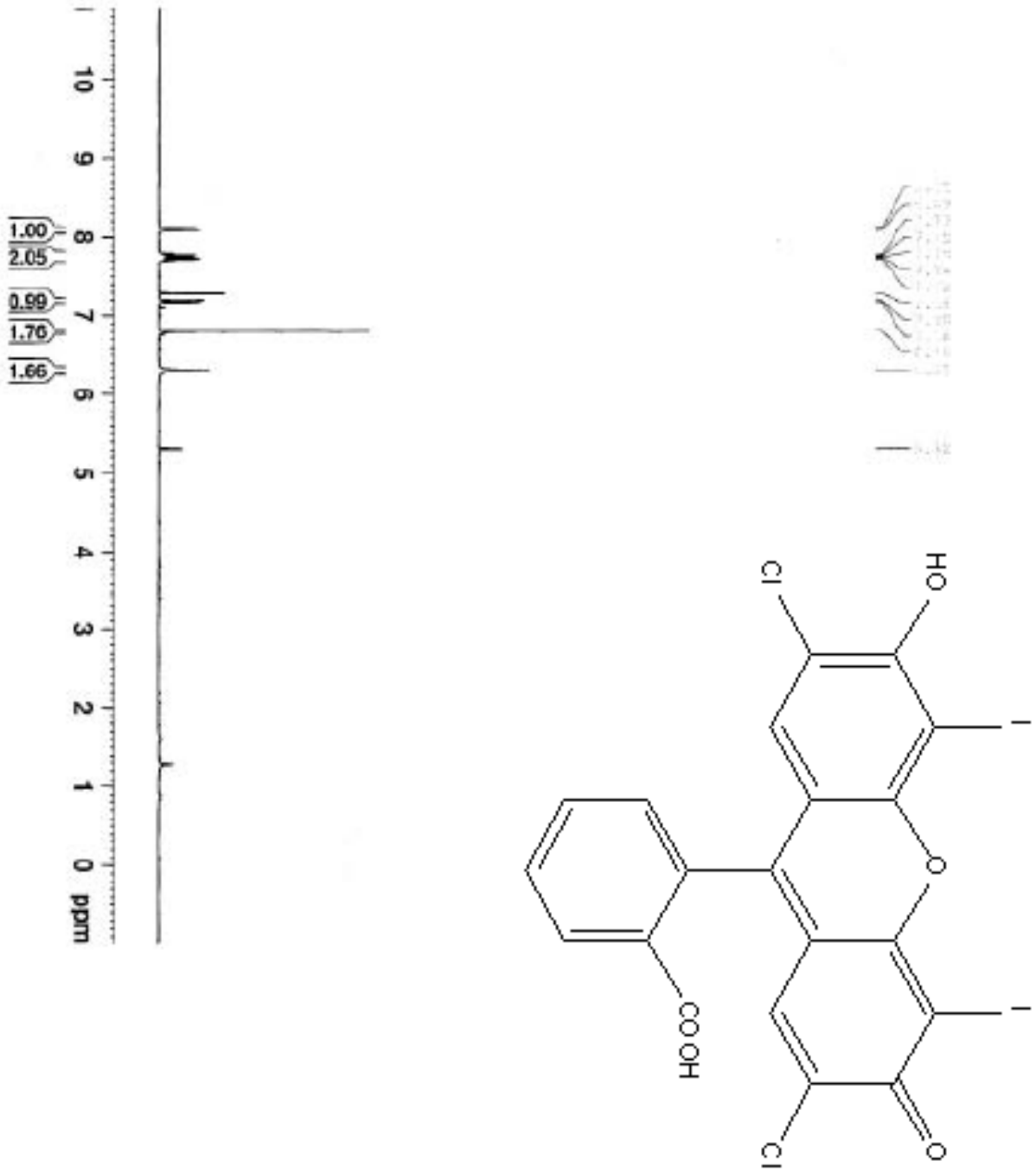

I
I
Br
OH
1.00
0.99
0.90
11
10
9
8
7
6
5
4
3
2
1
0
ppm


Br
OH
2.15
1.00
ppm
0
1
2
3
4
5
6
7
8
9
10
11


1.00
0.94
0.95
0.82
11
10
9
8
7
6
5
4
3
2
1
0
ppm
HO
HO


1.00
2.06
11
10
9
8
7
6
5
4
3
2
1
0
ppm
HO
I
I
I
HO
7.95
7.28
5.89


HO
I
I
OH
I
OH
3.00
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
5.75
4.05
4.02
3.32
2.50
1.99
1.20
1.18
1.16
1.09


1.00
2.05
0.99
1.70
1.66
10
9
8
7
6
5
4
3
2
1
0
ppm
Cl
HO
I
O
I
CO OH
Cl
O