SUPPORTING INFORMATION

For the

Communication entitled

**Effect of Base for the Efficient Synthesis of 4-Formylcoumarins and 4-
Formylcarbostyrils**

Authored by

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Experimental Section

General experimental information

Melting points were recorded on a Sheetal Scientific instrument and presented in degrees centigrade. IR spectra were recorded on a Nicolet 410 Fourier transform (FT) infrared spectrometer. The NMR was recorded on Bruker 400-MHz FT NMR spectrometers in DMSO- d_6 . ^1H and ^{13}C chemical shifts were represented as δppm values relative to the internal standard tetramethylsilane.

General procedure for the synthesis of 2 and 4: The substituted 4-bromomethylcoumarins/4-bromomethylcarbostyrils **1/3** (0.5gm; 1.0 equiv.) was heated with dimethylsulphoxide (2 mL) for 30 min at 70 $^{\circ}\text{C}$, it forms clear solution. Cooled the reaction at room temperature and added 5% sodium carbonate solution (2 mL) into the reaction mixture, continue the heating at 110 $^{\circ}\text{C}$ on oil bath for 6–16 h and reaction was monitored by TLC. After completion, the reaction was cooled and diluted with ice cooled water (20 mL), the solid separated was filtered, and washed with water, and no further purification is required.

4-Formyl-6-methylcoumarin 2a: Isolated compound light yellow solid; mp: 164-166 °C ; IR (KBr) ν 1736, 1703 cm^{-1} ; ^1H NMR (400MHz, DMSO- d_6) δ 10.14 (s,1H), 8.27 (s,1H) 7.50 (dd, 1H, J =1.6 Hz and 8.8 Hz), 7.37(d, 1H, J = 8.8 Hz), 7.13(s,1H), 2.38(s, 3H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 193, 160.08, 151.99, 143.36, 134.14, 133.20, 125.45, 124.77, 116.48, 114.65, 20.48; m/z (GC-MS) 188 (Calcd 188).

4-Formyl-7-methylcoumarin 2b: Isolated compound light yellow solid; mp: 150-152 °C IR (KBr) ν 1728, 1710 cm^{-1} ; ^1H NMR (400MHz, DMSO- d_6) δ 10.18 (s,1H), 8.48 (s,1H) 7.61(dd, 1H, J =1.4 Hz and 8.2 Hz), 7.42(d, 1H, J = 8.2 Hz), 7.20(s,1H), 2.41(s, 3H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 193.01, 160.10, 151.93, 143.41, 134.13, 133.22, 125.43, 124.72, 116.52, 114.51, 20.41; m/z (GC-MS) 188 (Calcd 188).

4-Formyl-6-methoxycoumarin 2c: Isolated compound yellow solid; mp: 168-170 °C; IR(KBr) ν 1706,1666; cm^{-1} ; ^1H NMR (400MHz, DMSO- d_6) δ 10.15(s,1H), 7.99(d, 1H, J =

2.8 Hz), 7.44(d, 1H, $J = 8.8\text{Hz}$), 7.30 (dd, 1H, $J = 2.8\text{ Hz and } 4.8\text{ Hz}$), 7.17(s,1H), 3.81(s, 3H); ^{13}C NMR(100MHz,DMSO- d_6) δ 193.55, 160.06, 155.79, 148.25, 142.99, 125.33, 119.56, 117.73, 115.31, 108.48, 55.68; m/z (GC-MS) 204 (calcd 204).

4-Formyl-6-chlorocoumarin 2d: Isolated compound pale yellow solid; mp: 180-184 $^{\circ}\text{C}$; IR(KBr) ν 1734,1708 cm^{-1} ; ^1H NMR (400MHz, DMSO- d_6) δ 10.116(s,1H), 8.48(d, 1H, $J = 2.4\text{ Hz}$),7.74(dd, 1H, $J = 2.40\text{Hz and } 9.2\text{ Hz}$) ,7.53(d,1H, $J = 8.80\text{ Hz}$), 7.26(s,1H); ^{13}C NMR (100MHz, DMSO- d_6) δ 193.06, 159.51, 152.44, 142.22, 131.97, 128.75, 126.08, 125.01, 118.71, 116.26; m/z (GC-MS) 208(calcd 208).

4-Formyl-6-bromocoumarin 2e: Isolated compound pale yellow solid; mp: 196-198 $^{\circ}\text{C}$; IR(KBr) ν 1733, 1707 cm^{-1} ; ^1H NMR (400 MHz, DMSO- d_6) δ 10.109, (s,1H), 8.614(d, 1H, $J = 2.40\text{ Hz}$), 7.85(dd,1H, $J = 2.40\text{ Hz and } 8.8\text{ Hz}$), 7.46(dd,1H, $J = 8.80\text{ Hz}$), 7.25(s, 1H); ^{13}C NMR (100MHz, DMSO- d_6) δ 193.61, 160.00, 153.39, 142.70, 135.31, 128.51, 126.59, 119.54, 117.28, 117.17; m/z (GC-MS) 252 (calcd. 252).

4-Formyl-5,6-benzocoumarin 2f: Isolated compound yellow solid; mp: 112-114 °C; IR(KBr) ν 1726, 1708, cm^{-1} ; ^1H NMR(400MHz, DMSO- d_6) δ 10.21(s, 1H), 8.46(d, 1H, $J = 8.8$ Hz), 8.39(d, 1H, $J = 8.4$ Hz), 8.04 (d, 1H, $J = 8.4$ Hz), 7.89(d, 1H, $J = 8.8$ Hz), 7.73(m, 2H), 7.25(s, 1H); ^{13}C -NMR (100MHz, DMSO- d_6) δ 193.46, 159.90, 151.00, 144.33, 134.22, 129.14, 127.51, 124.43, 123.78, 122.09, 121.59, 121.05, 110.82; m/z (GC-MS)224 (calcd. 224).

4-Formyl-7,8-benzocoumarin 2g: Isolated compound light pink solid; mp: 200-202 °C; IR(KBr) ν 1718, 1711, cm^{-1} ; ^1H NMR(400MHz, DMSO- d_6) δ 10.25(s, 1H), 8.39(d, 1H, $J = 8.6$ Hz), 8.32(d, 1H, $J = 8.42$ Hz), 8.11 (d, 1H, $J = 8.41$ Hz), 7.78(d, 1H, $J = 8.7$ Hz), 7.68(m, 2H), 7.19(s, 1H); ^{13}C -NMR (100MHz, DMSO- d_6) δ 193.41, 159.88, 151.09, 144.36, 134.19, 129.16, 127.48, 124.41, 123.75, 122.05, 121.56, 121.08, 110.85; m/z (GC-MS)224 (calcd. 224).

4-Formyl-5,7-dimethylcoumarin 2h: Isolated compound light yellow solid; mp: 138-134 °C; IR (KBr) ν 1725, 1710 cm^{-1} ; ^1H NMR (400MHz, DMSO- d_6) δ 10.11(s,1H), 7.74 (s,1H) 7.53(1H), 7.14(s,1H), 2.72(s, 3H), 2.36(s,3 H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 191.08, 160.33, 155.94, 151.83, 142.11, 135.91, 125.98, 116.42, 115.25, 114.97, 23.89, 21.11; m/z (GC-MS) 188 (Calcd 188).

4-Formyl-7-hydroxycoumarin 2i: Isolated compound colorless solid; mp: 128-130 °C; IR(KBr) ν 3414, 1699, 1680; cm^{-1} ; ^1H NMR (400MHz, DMSO- d_6) δ 10.21(s,1H), 7.89(d, 1H, $J = 2.8$ Hz), 7.56(d, 1H, $J = 8.8$ Hz), 7.41(dd, 1H, $J = 2.7$ Hz and 8.6 Hz), 7.11(s,1H), 3.48(br, s, 1H D $_2$ O Exchangeable); ^{13}C NMR(100MHz,DMSO- d_6) δ 192.88, 161.11, 156.62, 147.91, 143.01, 124.87, 120.02, 117.93, 116.11, 110.12; m/z(GC-MS) 190(calcd 190).

4-Formylcarboistyril 4a: Isolated compound yellow solid; mp: 214-216 °C; IR(KBr) ν 3404, 1709,1663 cm^{-1} ; ^1H NMR(400MHz, DMSO- d_6) δ 12.14 (br s, 1H, NH), 10.17 (s. 1H), 8.56 (d, 1H, $J = 8.0$ Hz), 7.58(t, 1H), 9.39(d, 1H, $J = 7.6$ Hz), 7.27 (t, 1H), 7.71(s, 1H); NMR (100MHz, DMSO- d_6) δ 194.69, 161.32, 141.15, 139.58, 130.98, 130.40, 130.17, 125.98, 124.62, 115.39; m/z (GC-MS) 207 (Calcd 207).

4-Formyl-6-chlorocarbostyryl 4b: Isolated compound white solid; mp: 220-222 °C; IR(KBr) ν 3152, 1703, 1672 cm^{-1} ; ^1H NMR(400MHz,DMSO- d_6) δ 12.26 (br s, 1H, NH), 10.11 (s, 1H), 8.55 (d, 1H, J = 2.0 Hz), 7.61 (d 1H, J = 6.4 Hz), 7.38 (d, 1H, J = 5.2 Hz), 7.27 (s, 1H); ^{13}C NMR(100MHz,DMSO- d_6), δ 194.96, 161.82, 147.61, 140.54, 138.85, 133.42, 131.40, 127.22, 124.81, 117.98; m/z (GC-MS) 173 (Calcd 173).

4-Formyl-6-bromocarbostyryl 4c: Isolated compound yellow solid; mp:198-200 °C; IR(KBr) ν 3236, 1710, 1682 cm^{-1} ; ^1H NMR(400MHz,DMSO- d_6) δ 12.31 (br s, 1H, NH), 10.16 (s, 1H), 8.67 (d, 1H, J = 2.3 Hz), 7.68 (d 1H, J = 8.1Hz), 7.49 (d, 1H, J = 7.4 Hz), 7.30 (s, 1H); ^{13}C NMR(100MHz,DMSO- d_6), δ 195.41, 162.73, 148.18, 141.11, 139.21, 134.31, 131.96, 127.86, 125.01, 118.18; m/z (GC-MS) 252 (Calcd 252).

4-Formyl-6-methylcarbostyryl 4d: Isolated compound light yellow solid; mp: 175-177 °C IR(KBr) ν 3272, 1716,1671 cm^{-1} ; ^1H NMR(400MHz, DMSO- d_6) δ 10.14 (s, 1H), 8.38 (s, 1H), 7.46 (dd, 1H, J =1.8 Hz and 6.8 Hz), 7.28(d, 1H, J = 6.8 Hz), 7.16(s, 1H), 2.38(s, 3H); ^{13}C

NMR (100 MHz, DMSO- d_6) δ 194.01, 161.10, 152.01, 142.98, 133.68, 133.85, 125.88, 124.67, 117.13, 116.11, 20.18; m/z (GC-MS) 187 (Calcd 187).

Table 3: Melting points of Compounds **2a** to **2i** and **4a** to **4d**

Entry	Mp (°C)	Mp (°C)	Mp (°C)
	*Our report	Literature reported (ChemSpider)	Literature reported (Scifinder)
2a	164-166	172.385	168-169
		Data Source: J. Heterocycl. Chem. 2006, 43(5), 1367-1369	Data Sources: Ind. J. Chem. B: 1982, V21B 5, 399 402.
2b	181-183	174.072	201-202 ; 185-187
		Data Sources: J. Heterocycl. Chem. 2006, 43(5), 1367-1369	Data Sources: (1) Eur. J. Org. Chem. 2007, 26, 4301-4304; (2) Ind. J. Chem. Section B:1982, V21B 5, 399-402.
2c	168-170	-	192.7±28.8
			Data Sources:
2d	180-184	192.4±27.7	184-186
		Data Sources: Cambridge Crystallographic data centre	Data Sources: Cryst. Eng. Comm. 2003, V5, 507-513.
2e	196-198	-	209.3±28.7
			Data Sources:
2f	112-114	-	215.1±27.4

			Data Sources:
2g	200-202	215.102	215.1±27.4
	Data Sources: J. Heterocycl. Chem. 2006, 43(5), 1367-1369		Data Sources:
2h	138-134	152	165-166
	Data Source:		Data Sources: J. Heterocycl. Chem. 1988, 25, 6, 1681.
2i	128-130	-	223-225
			Data Sources: Eur. J. Org. Chem. 2007, 26, 4301-4304.
4a	214-216	203.231	267, 261-262, 257
	Data Sources: ABI Chemicals, External id; AC2A03D96, Biochemistry 1996 , 35, 4457-4467		Data Sources: (1) AIST: Integrated Spectral Database System of Organic Compounds. (Data were obtained from the National Institute of Advanced Industrial Science and Technology (Japan)). (2) Indiana Academy of Science 1961, V70, P115-18. (3) Monatshefte fuer Chemie 1966, V97 (2), P409-16.
4b	220-222	228.374	228.4±28.7
	Data Sources: J. Heterocycl. Chem. 1972, <u>9(1)</u> , 175-176.		Data Sources: (1) Calculated using Advanced Chemistry

				Development (ACD/Labs) Software V11.02 (© 1994-2014 ACD/Labs).(2) Predicted NMR data calculated using Advanced Chemistry Development, Inc. (ACD/Labs) Software V11.01 (© 1994-2014 ACD/Labs).
4c	198-200	-	235.6±28.7	Data Sources: (1) Calculated using Advanced Chemistry Development (ACD/Labs) Software V11.02 (© 1994-2014 ACD/Labs). (2) Predicted NMR data calculated using Advanced Chemistry Development, Inc. (ACD/Labs) Software V11.01 (© 1994-2014 ACD/Labs).
4d	175-177	210.314	193.8±28.9	
		Data Sources: AKOs, External ID;AKI-BBV-00014126, Innovapharm, External id; BBV-00014126.	Data Sources:	

** Note: Melting points were recorded on a Sheetal Scientific instrument (hot plate system)*

Spectrum No. 1: ^1H NMR of Compound **2a** in DMSO-d_6

Spectrum No. 2: ^{13}C NMR of Compound **2a** in DMSO-d_6

Spectrum No. 3: ^1H NMR of Compound **2c** in DMSO-d_6

Spectrum No. 4: ^{13}C NMR of Compound **2c** in DMSO-d_6

Spectrum No. 5: ^1H NMR of Compound **2d** in DMSO_d6

Spectrum No. 6: ^{13}C NMR of Compound **2d** in DMSO_d6

Spectrum No. 7: ^1H NMR of Compound **2e** in DMSO-d_6

Spectrum No. 8: ^{13}C NMR of Compound **2e** in DMSO-d_6

Spectrum No. 9: ^1H NMR of Compound **2g** in DMSO-d_6

Spectrum No. 10: ^{13}C NMR of Compound **2g** in DMSO-d_6

Spectrum No. 11: ^1H NMR of Compound **2i** in DMSO-d_6

Spectrum No. 12: ^{13}C NMR of Compound **2i** in DMSO-d_6

Current Data Parameters
NAME g1_megharaj
EXPNO 44
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140212
Time 4.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG sg
TD 16384
SOLVENT DMSO
NS 8
DS 0
SWH 6410.286 Hz
FIDRES 0.391251 Hz
AQ 1.2779520 sec
RG 203
DW 78.000 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.10 usec
PL1 -3.00 dB
PL1W 13.42244530 W
SFO1 400.2220017 MHz

F2 - Processing parameters
SI 262144
SF 400.2200098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Spectrum No. 13: ^1H NMR of Compound **4a** in DMSO-d_6

Current Data Parameters
NAME g1_megharaj
EXPNO 45
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140212
Time 4.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 1332
DS 0
SWH 24038.461 Hz
FIDRES 0.732596 Hz
AQ 0.6815744 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 3.50000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.62 usec
PL1 -2.00 dB
PL1W 58.25462341 W
SFO1 100.6264810 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 50.00 usec
PL2 -3.00 dB
PL12 13.10 dB
PL13 16.00 dB
PL2W 13.42244530 W
PL12W 0.32948193 W
PL13W 0.16897857 W
SFO2 400.2316009 MHz

F2 - Processing parameters
SI 32768
SF 100.6279688 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

Spectrum No. 14: ^{13}C NMR of Compound **4a** in DMSO-d_6

Spectrum No. 13: ^1H NMR of Compound **4b** in DMSO-d_6

Spectrum No. 14: ^{13}C NMR of Compound **4b** in DMSO-d_6

Crystal Structure Report of Compound **2a**

A specimen of $\text{C}_{11}\text{H}_8\text{O}_3$ was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

The total exposure time was 1.30 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 5103 reflections to a maximum θ angle of 29.80° (0.72 Å resolution), of which 2250 were independent (average redundancy 2.268,

completeness = 88.4%, $R_{\text{int}} = 3.09\%$, $R_{\text{sig}} = 3.72\%$) and 1442 (64.09%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 12.7158(9) \text{ \AA}$, $b = 9.0033(6) \text{ \AA}$, $c = 7.7537(5) \text{ \AA}$, $\beta = 90.176(4)^\circ$, volume = $887.67(10) \text{ \AA}^3$, are based upon the refinement of the XYZ-centroids of 1621 reflections above $20 \sigma(I)$ with $5.544^\circ < 2\theta < 56.96^\circ$. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.891.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group $P 1 21/c 1$, with $Z = 4$ for the formula unit, $C_{11}H_8O_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 129 variables converged at $R1 = 11.14\%$, for the observed data and $wR2 = 42.96\%$ for all data. The goodness-of-fit was 1.177. The largest peak in the final difference electron density synthesis was $0.296 \text{ e}^-/\text{\AA}^3$ and the largest hole was $-0.290 \text{ e}^-/\text{\AA}^3$ with an RMS deviation of $0.085 \text{ e}^-/\text{\AA}^3$. On the basis of the final model, the calculated density was 1.408 g/cm^3 and $F(000)$, 392 e^- .

Table 1. Sample and crystal data for **2a**:

Identification code	Chem_Megharaj_SI69_1	
Chemical formula	C ₁₁ H ₈ O ₃	
Formula weight	188.17	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 12.7158(9) Å	α = 90°
	b = 9.0033(6) Å	β = 90.176(4)°
	c = 7.7537(5) Å	γ = 90°
Volume	887.67(10) Å ³	
Z	4	
Density (calculated)	1.408 Mg/cm ³	
Absorption coefficient	0.103 mm ⁻¹	
F(000)	392	

Table 2. Data collection and structure refinement for **2a**

Theta range for data collection	1.60 to 29.80°	
Index ranges	-17<= <i>h</i> <=14, -9<= <i>k</i> <=12, -10<= <i>l</i> <=10	
Reflections collected	5103	
Independent reflections	2250 [R(int) = 0.0309]	
Coverage of independent reflections	88.4%	
Absorption correction	multi-scan	
Structure solution technique	direct methods	

Structure solution program	SHELXS-97 (Sheldrick, 2008)	
Refinement method	Full-matrix least-squares on F ²	
Refinement program	SHELXL-97 (Sheldrick, 2008)	
Function minimized	$\sum w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	2250 / 0 / 129	
Goodness-of-fit on F²	1.177	
Δ/σ_{max}	0.181	
Final R indices	1442 data; $I > 2\sigma(I)$	R1 = 0.1114, wR2 = 0.4169
	all data	R1 = 0.1384, wR2 = 0.4296
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.1577P)^2 + 2.2740P]$ where $P = (F_o^2 + 2F_c^2)/3$	
Extinction coefficient	0.0230(100)	
Largest diff. peak and hole	0.296 and -0.290 eÅ ⁻³	
R.M.S. deviation from mean	0.085 eÅ ⁻³	

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å²) for **2a**

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
O1	0.9586(3)	0.7521(4)	0.2623(7)	0.0647(13)
O2	0.0828(3)	0.8765(4)	0.1305(5)	0.0473(10)
O3	0.3033(4)	0.4395(5)	0.9164(6)	0.0719(14)
C1	0.0369(4)	0.7458(5)	0.1776(7)	0.0450(11)
C2	0.1750(4)	0.8811(5)	0.0375(6)	0.0413(11)
C3	0.2132(5)	0.0217(5)	0.0016(7)	0.0512(13)
C4	0.3065(5)	0.0346(6)	0.9128(7)	0.0545(14)
C5	0.3614(4)	0.9108(7)	0.8573(7)	0.0528(13)
C6	0.4637(5)	0.9255(8)	0.7580(9)	0.0732(19)
C7	0.3207(4)	0.7708(5)	0.8935(7)	0.0453(12)
C8	0.2267(4)	0.7525(4)	0.9842(6)	0.0369(10)
C9	0.1801(4)	0.6126(5)	0.0329(6)	0.0393(10)
C10	0.2247(5)	0.4647(6)	0.9911(8)	0.0549(14)
C11	0.0900(4)	0.6117(5)	0.1244(6)	0.0422(11)

Table 4. Bond lengths (Å) for **2a**

O1-C1	1.196(6)	O2-C1	1.363(6)
O2-C2	1.379(6)	O3-C10	1.179(7)
C1-C11	1.445(7)	C2-C3	1.385(6)
C2-C8	1.395(6)	C3-C4	1.378(8)
C3-H3	0.93	C4-C5	1.384(8)
C4-H4	0.93	C5-C7	1.391(7)
C5-C6	1.520(8)	C6-H6A	0.96

C6-H6B	0.96	C6-H6C	0.96
C7-C8	1.399(7)	C7-H7	0.93
C8-C9	1.442(6)	C9-C11	1.349(6)
C9-C10	1.484(7)	C10-H10	0.93
C11-H11	0.93		

Table 5. Bond angles (°) for 2a

C1-O2-C2	122.1(4)	O1-C1-O2	117.7(4)
O1-C1-C11	126.0(5)	O2-C1-C11	116.3(4)
O2-C2-C3	115.6(4)	O2-C2-C8	122.2(4)
C3-C2-C8	122.2(5)	C4-C3-C2	118.7(5)
C4-C3-H3	120.6	C2-C3-H3	120.6
C3-C4-C5	121.6(5)	C3-C4-H4	119.2
C5-C4-H4	119.2	C4-C5-C7	118.6(5)
C4-C5-C6	121.4(5)	C7-C5-C6	120.0(6)
C5-C6-H6A	109.5	C5-C6-H6B	109.5
H6A-C6-H6B	109.5	C5-C6-H6C	109.5
H6A-C6-H6C	109.5	H6B-C6-H6C	109.5
C5-C7-C8	121.8(5)	C5-C7-H7	119.1
C8-C7-H7	119.1	C7-C8-C2	117.1(4)
C7-C8-C9	126.0(4)	C2-C8-C9	116.9(4)

C11-C9-C8	119.5(4)	C11-C9-C10	115.8(4)
C8-C9-C10	124.7(5)	O3-C10-C9	127.2(5)
O3-C10-H10	116.4	C9-C10-H10	116.4
C9-C11-C1	123.0(4)	C9-C11-H11	118.5
C1-C11-H11	118.5		

Table 6. Anisotropic atomic displacement parameters (Å²) for **2a**

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
O1	0.058(3)	0.057(2)	0.079(3)	0.0034(18)	0.029(2)	0.0035(17)
O2	0.049(2)	0.0402(17)	0.053(2)	0.0018(14)	0.0120(17)	0.0016(14)

O3	0.068(3)	0.054(2)	0.094(3)	-0.011(2)	0.030(3)	0.013(2)
C1	0.045(3)	0.045(2)	0.045(3)	0.0020(19)	0.007(2)	0.0018(19)
C2	0.047(3)	0.040(2)	0.037(2)	-0.0003(17)	0.008(2)	0.0004(18)
C3	0.065(3)	0.036(2)	0.053(3)	-0.003(2)	0.007(3)	-0.002(2)
C4	0.064(3)	0.045(3)	0.055(3)	0.005(2)	0.003(3)	-0.019(2)
C5	0.041(2)	0.071(3)	0.046(3)	0.004(2)	0.002(2)	-0.016(2)
C6	0.062(4)	0.083(5)	0.074(4)	0.006(3)	0.019(4)	-0.018(3)
C7	0.040(2)	0.051(3)	0.045(3)	-0.002(2)	0.004(2)	-0.0008(19)
C8	0.041(2)	0.035(2)	0.035(2)	0.0012(15)	0.0022(19)	0.0001(16)
C9	0.045(2)	0.034(2)	0.039(2)	-0.0006(16)	0.005(2)	0.0018(17)
C10	0.065(3)	0.035(2)	0.064(3)	-0.001(2)	0.010(3)	0.002(2)
C11	0.043(2)	0.037(2)	0.047(2)	-0.0008(18)	0.006(2)	-0.0036(18)

Table 7. Hydrogen atomic coordinates and isotropic atomic displacement parameters ($\text{\AA}^2$) for **2a**

	x/a	y/b	z/c	U(eq)
H3	0.1767	1.1059	0.0369	0.061
H4	0.3331	1.1286	-0.1104	0.065
H6A	0.4486	0.9501	-0.3600	0.11

H6B	0.5014	0.8332	-0.2374	0.11
H6C	0.5058	1.0027	-0.1914	0.11
H7	0.3570	0.6872	-0.1437	0.054
H10	0.1864	0.3827	0.0280	0.066
H11	0.0609	0.5207	0.1546	0.051

Crystal Structure Report of Compound 2c

A specimen of $C_{11}H_8O_4$ was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

Table 1: Data collection details for **2c**.

Axis	dx/mm	2 θ /°	ω /°	ϕ /°	χ /°	Width/ °	Frames	Time/ s	Wavelength/ Å	Voltage/ kV	Current/ mA	Temp./ K
Phi	60.407	-65.00	-155.00	-360.00	54.74	0.50	739	5.00	0.71073	20	5.0	n/a
Omega	60.407	-62.50	101.96	-67.50	54.74	0.50	282	5.00	0.71073	20	5.0	n/a
Omega	60.407	-60.00	-193.78	-205.00	54.74	0.50	302	5.00	0.71073	20	5.0	n/a
Omega	60.407	-30.00	-230.58	-132.50	54.74	0.50	218	5.00	0.71073	20	5.0	n/a
Phi	60.407	-27.50	-90.00	3.93	54.74	0.50	313	5.00	0.71073	20	5.0	n/a
Omega	60.407	-25.00	-225.76	-227.50	54.74	0.50	252	5.00	0.71073	20	5.0	n/a
Omega	60.407	2.50	-168.07	-95.00	54.74	0.50	269	5.00	0.71073	12	1.0	n/a

Phi	60.407	10.00	-50.00	-360.00	54.74	0.50	739	5.00	0.71073	34	25.0	n/a
Omega	60.407	55.00	-13.34	-17.50	54.74	0.50	168	5.00	0.71073	50	30.0	n/a
Phi	60.407	62.50	35.00	8.74	54.74	0.50	173	5.00	0.71073	50	30.0	n/a

A total of 3455 frames were collected. The total exposure time was 4.80 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 21859 reflections to a maximum θ angle of 40.90° (0.54 Å resolution), of which 5921 were independent (average redundancy 3.692, completeness = 98.8%, $R_{\text{int}} = 2.82\%$, $R_{\text{sig}} = 3.78\%$) and 2743 (46.33%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 13.1460(9)$ Å, $b = 15.5498(10)$ Å, $c = 9.0381(6)$ Å, $\beta = 98.535(4)^\circ$, volume = $1827.1(2)$ Å³, are based upon the refinement of the XYZ-centroids of 4085 reflections above $20 \sigma(I)$ with $5.239^\circ < 2\theta < 67.12^\circ$. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.928.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group $C 1 2/c 1$, with $Z = 8$ for the formula unit, $C_{11}H_8O_4$. The final anisotropic full-matrix least-squares refinement on F^2 with 138 variables converged at $R1 = 6.06\%$, for the observed data and $wR2 = 20.87\%$ for all data. The goodness-of-fit was 1.014. The largest peak in the final difference electron density synthesis was $0.280 \text{ e}^-/\text{\AA}^3$ and the largest hole was $-0.315 \text{ e}^-/\text{\AA}^3$ with an RMS deviation of $0.128 \text{ e}^-/\text{\AA}^3$. On the basis of the final model, the calculated density was 1.485 g/cm^3 and $F(000)$, 848 e^- .

Table 2. Sample and crystal data for **2c**.

Identification code	CHEM_MANJUNATH_SI117	
Chemical formula	C ₁₁ H ₈ O ₄	
Formula weight	204.17	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	C 1 2/c 1	
Unit cell dimensions	a = 13.1460(9) Å	α = 90°
	b = 15.5498(10) Å	β = 98.535(4)°
	c = 9.0381(6) Å	γ = 90°
Volume	1827.1(2) Å ³	
Z	8	
Density (calculated)	1.485 Mg/cm ³	
Absorption coefficient	0.114 mm ⁻¹	
F(000)	848	

Table 3. Data collection and structure refinement for **2c**.

Theta range for data collection	2.04 to 40.90°	
Index ranges	-23<=h<=20, -28<=k<=27, -16<=l<=16	
Reflections collected	21859	
Independent reflections	5921 [R(int) = 0.0282]	
Coverage of independent reflections	98.8%	
Absorption correction	multi-scan	
Structure solution technique	direct methods	
Structure solution program	SHELXS-97 (Sheldrick, 2008)	
Refinement method	Full-matrix least-squares on F ²	
Refinement program	SHELXL-97 (Sheldrick, 2008)	
Function minimized	$\sum w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	5921 / 0 / 138	
Goodness-of-fit on F²	1.014	
$\Delta/\sigma_{\max}$	0.091	
Final R indices	2743 data; $I > 2\sigma(I)$	R1 = 0.0606, wR2 = 0.1702
	all data	R1 = 0.1308, wR2 =

		0.2087
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0996P)^2+0.0398P]$ where $P=(F_o^2+2F_c^2)/3$	
Extinction coefficient	0.0042(10)	
Largest diff. peak and hole	0.280 and -0.315 eÅ ⁻³	
R.M.S. deviation from mean	0.128 eÅ ⁻³	

Table 4. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å²) for **2c**.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
O1	0.87469(7)	0.56420(5)	0.94207(9)	0.0561(3)
O2	0.88682(6)	0.91480(4)	0.01139(8)	0.0448(2)
O3	0.87563(8)	0.03529(6)	0.13406(9)	0.0665(3)
O4	0.85360(7)	0.70148(5)	0.43276(9)	0.0603(3)
C1	0.87165(11)	0.50688(7)	0.06476(16)	0.0641(4)

C2	0.88099(8)	0.65021(6)	0.97265(11)	0.0398(2)
C3	0.87587(7)	0.68533(6)	0.11121(10)	0.0367(2)
C4	0.87809(6)	0.77511(6)	0.12960(9)	0.03202(18)
C5	0.88638(7)	0.82606(6)	0.00494(10)	0.03529(19)
C6	0.87787(9)	0.95817(7)	0.14017(10)	0.0440(2)
C7	0.87160(8)	0.90632(6)	0.27218(10)	0.0397(2)
C8	0.87070(7)	0.81987(6)	0.26841(9)	0.03363(19)
C9	0.86016(8)	0.77667(7)	0.41255(10)	0.0427(2)
C10	0.89339(8)	0.79039(7)	0.86595(10)	0.0421(2)
C11	0.89024(8)	0.70320(7)	0.85003(11)	0.0434(2)

Table 5. Bond lengths (Å) for **2c**.

O1-C2	1.3657(11)	O1-C1	1.4279(14)
O2-C6	1.3654(12)	O2-C5	1.3811(10)
O3-C6	1.2006(14)	O4-C9	1.1884(12)
C1-H1A	0.96	C1-H1B	0.96
C1-H1C	0.96	C2-C3	1.3770(13)
C2-C11	1.4007(14)	C3-C4	1.4058(12)
C3-H3	0.93	C4-C5	1.3948(12)
C4-C8	1.4504(11)	C5-C10	1.3887(12)
C6-C7	1.4524(13)	C7-C8	1.3449(12)
C7-H7	0.93	C8-C9	1.4903(11)
C9-H9	0.93	C10-C11	1.3634(13)
C10-H10	0.93	C11-H11	0.93

Table 6. Bond angles (°) for **2c**.

C2-O1-C1	117.52(8)	C6-O2-C5	121.95(7)
O1-C1-H1A	109.5	O1-C1-H1B	109.5
H1A-C1-H1B	109.5	O1-C1-H1C	109.5
H1A-C1-H1C	109.5	H1B-C1-H1C	109.5

O1-C2-C3	124.25(9)	O1-C2-C11	115.19(8)
C3-C2-C11	120.53(9)	C2-C3-C4	119.90(9)
C2-C3-H3	120.1	C4-C3-H3	120.1
C5-C4-C3	118.12(8)	C5-C4-C8	116.71(8)
C3-C4-C8	125.16(8)	O2-C5-C10	115.90(8)
O2-C5-C4	122.26(8)	C10-C5-C4	121.84(8)
O3-C6-O2	117.31(8)	O3-C6-C7	126.03(9)
O2-C6-C7	116.66(9)	C8-C7-C6	122.38(8)
C8-C7-H7	118.8	C6-C7-H7	118.8
C7-C8-C4	120.00(8)	C7-C8-C9	115.47(8)
C4-C8-C9	124.52(8)	O4-C9-C8	126.58(9)
O4-C9-H9	116.7	C8-C9-H9	116.7
C11-C10-C5	119.18(9)	C11-C10-H10	120.4
C5-C10-H10	120.4	C10-C11-C2	120.42(9)
C10-C11-H11	119.8	C2-C11-H11	119.8

Table 7. Anisotropic atomic displacement parameters (Å²) for **2c**.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
O1	0.0792(6)	0.0381(4)	0.0514(5)	-0.0130(3)	0.0110(4)	-0.0016(4)
O2	0.0689(5)	0.0353(3)	0.0306(3)	0.0030(2)	0.0090(3)	0.0031(3)
O3	0.1204(9)	0.0331(4)	0.0449(4)	0.0024(3)	0.0088(5)	0.0039(4)
O4	0.0971(7)	0.0429(4)	0.0449(4)	0.0087(3)	0.0239(4)	-0.0019(4)
C1	0.0860(10)	0.0357(5)	0.0676(8)	-0.0018(5)	0.0019(7)	0.0002(6)
C2	0.0421(5)	0.0374(5)	0.0398(5)	-0.0082(4)	0.0058(4)	0.0003(4)
C3	0.0422(5)	0.0350(4)	0.0332(4)	-0.0011(3)	0.0064(3)	-0.0001(3)
C4	0.0348(4)	0.0342(4)	0.0272(3)	-0.0009(3)	0.0050(3)	0.0014(3)
C5	0.0417(5)	0.0358(4)	0.0287(4)	0.0007(3)	0.0061(3)	0.0016(3)

C6	0.0646(7)	0.0334(4)	0.0331(4)	0.0009(3)	0.0042(4)	0.0028(4)
C7	0.0549(6)	0.0357(4)	0.0286(4)	-0.0022(3)	0.0068(4)	0.0042(4)
C8	0.0397(5)	0.0349(4)	0.0267(3)	0.0003(3)	0.0062(3)	0.0013(3)
C9	0.0586(6)	0.0416(5)	0.0298(4)	0.0016(3)	0.0126(4)	0.0008(4)
C10	0.0520(6)	0.0476(5)	0.0277(4)	0.0002(3)	0.0093(4)	0.0013(4)
C11	0.0490(6)	0.0499(6)	0.0322(4)	-0.0095(4)	0.0090(4)	0.0000(4)

Table 8. Hydrogen atomic coordinates and isotropic atomic displacement parameters ($\text{\AA}^2$) for **2c**.

	x/a	y/b	z/c	U(eq)
H1A	0.9330	0.5138	0.1359	0.096
H1B	0.8673	0.4487	0.0289	0.096
H1C	0.8126	0.5197	0.1119	0.096
H3	0.8709	0.6497	0.1925	0.044
H7	0.8681	0.9339	0.3626	0.048
H9	0.8586	0.8121	0.4950	0.051
H10	0.9002	0.8255	-0.2153	0.051
H11	0.8943	0.6788	-0.2428	0.052

Crystal Structure Report of Compound **2e**

A specimen of $C_{10}H_5BrO_3$ was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

Table 1: Data collection details for **2e**.

Axis	dx/ mm	2 θ / °	ω /°	ϕ /°	χ /°	Width h/°	Fra mes	Tim e/s	Wavelen gth/Å	Voltag e/kV	Current /mA	Temperat ure/K
Om	60.3	-	-	-	54.	0.50	323	5.00	0.71073	50	30.0	n/a
ega	68	30. 00	219. 21	20.0 0	74							
Om	60.3	32.	-	345.	54.	0.50	450	5.00	0.71073	50	30.0	n/a

ega	68	50	170. 00	00	74							
Om ega	60.3 68	35. 00	- 167. 50	247. 50	54. 74	0.50	295	5.00	0.71073	50	30.0	n/a
Om ega	60.3 68	40. 00	- 68.7 6	115. 00	54. 74	0.50	161	5.00	0.71073	50	30.0	n/a

A total of 1229 frames were collected. The total exposure time was 1.71 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 9164 reflections to a maximum θ angle of 29.46° ($0.72 \text{ \AA}$ resolution), of which 2439 were independent (average redundancy 3.757, completeness = 99.1%, $R_{\text{int}} = 3.70\%$, $R_{\text{sig}} = 3.88\%$) and 1652 (67.73%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 7.1969(3) \text{ \AA}$, $b = 8.5702(3) \text{ \AA}$, $c = 14.4945(5) \text{ \AA}$, $\beta = 96.561(2)^\circ$, volume = $888.15(6) \text{ \AA}^3$, are based upon the refinement of the XYZ-centroids of 2373 reflections above $20 \sigma(I)$ with $5.531^\circ < 2\theta < 47.63^\circ$. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.456.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group $P 1 21/n 1$, with $Z = 4$ for the formula unit, $C_{10}H_5BrO_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 127 variables converged at $R1 = 3.33\%$, for the observed data and $wR2 = 8.09\%$ for all data. The goodness-of-fit was 1.017. The largest peak in the final difference electron density synthesis was $0.366 \text{ e}^-/\text{\AA}^3$ and the

largest hole was $-0.407 \text{ e}^-/\text{\AA}^3$ with an RMS deviation of $0.064 \text{ e}^-/\text{\AA}^3$. On the basis of the final model, the calculated density was 1.892 g/cm^3 and $F(000)$, 496 e^- .

Table 2. Sample and crystal data for **2e**.

Identification code	Chem_Rashmi_SI188	
Chemical formula	C ₁₀ H ₅ BrO ₃	
Formula weight	253.05	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	P 1 21/n 1	
Unit cell dimensions	a = 7.1969(3) Å	α = 90°
	b = 8.5702(3) Å	β = 96.561(2)°
	c = 14.4945(5) Å	γ = 90°
Volume	888.15(6) Å ³	
Z	4	
Density (calculated)	1.892 Mg/cm ³	
Absorption coefficient	4.601 mm ⁻¹	

F(000)	496	
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Table 3. Data collection and structure refinement for **2e**.

Theta range for data collection	2.77 to 29.46°	
Index ranges	-9<= <i>h</i> <=9, -11<= <i>k</i> <=11, -19<= <i>l</i> <=20	
Reflections collected	9164	
Independent reflections	2439 [R(int) = 0.0370]	
Coverage of independent reflections	99.1%	
Absorption correction	multi-scan	
Structure solution technique	direct methods	
Structure solution program	SHELXS-97 (Sheldrick, 2008)	
Refinement method	Full-matrix least-squares on F ²	
Refinement program	SHELXL-97 (Sheldrick, 2008)	
Function minimized	$\sum w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	2439 / 0 / 127	

Goodness-of-fit on F2	1.017	
$\Delta/\sigma_{\max}$	0.009	
Final R indices	1652 data; $I > 2\sigma(I)$	R1 = 0.0333, wR2 = 0.0722
	all data	R1 = 0.0645, wR2 = 0.0809
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 0.0758P]$ where $P = (F_o^2 + 2F_c^2)/3$	
Largest diff. peak and hole	0.366 and -0.407 eÅ ⁻³	
R.M.S. deviation from mean	0.064 eÅ ⁻³	

Table 4. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å²) for **2e**.

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x/a	y/b	z/c	U(eq)
Br1	0.35590(4)	0.60100(3)	0.250065(17)	0.05739(13)

O1	0.2206(3)	0.60829(18)	0.83455(11)	0.0483(4)
O2	0.1333(3)	0.5033(2)	0.69796(11)	0.0588(5)
O3	0.1839(4)	0.1274(2)	0.03791(14)	0.0768(7)
C1	0.3184(3)	0.5989(3)	0.11864(15)	0.0400(5)
C2	0.3409(3)	0.7355(3)	0.07069(17)	0.0477(6)
C3	0.3079(4)	0.7353(3)	0.97541(17)	0.0477(6)
C4	0.2530(3)	0.5983(3)	0.93000(15)	0.0387(5)
C5	0.1601(3)	0.4823(3)	0.78036(17)	0.0450(6)
C6	0.2662(3)	0.4621(3)	0.07362(15)	0.0386(5)
C7	0.2312(3)	0.4592(3)	0.97700(14)	0.0341(5)
C8	0.1711(3)	0.3243(3)	0.92084(15)	0.0364(5)
C9	0.1454(4)	0.1663(3)	0.95971(18)	0.0495(6)
C10	0.1368(3)	0.3379(3)	0.82863(16)	0.0419(5)

Table 5. Bond lengths (Å) for **2e**.

Br1-C1	1.893(2)	O1-C5	1.376(3)
O1-C4	1.379(3)	O2-C5	1.201(3)
O3-C9	1.184(3)	C1-C6	1.373(3)
C1-C2	1.380(3)	C2-C3	1.374(3)
C2-H2	0.93	C3-C4	1.382(3)

C3-H3	0.93	C4-C7	1.390(3)
C5-C10	1.441(3)	C6-C7	1.395(3)
C6-H6	0.93	C7-C8	1.451(3)
C8-C10	1.336(3)	C8-C9	1.486(3)
C9-H9	0.93	C10-H10	0.93

Table 6. Bond angles (°) for **2e**.

C5-O1-C4	122.00(18)	C6-C1-C2	121.8(2)
C6-C1-Br1	119.08(17)	C2-C1-Br1	119.15(17)
C3-C2-C1	119.2(2)	C3-C2-H2	120.4
C1-C2-H2	120.4	C2-C3-C4	119.1(2)
C2-C3-H3	120.4	C4-C3-H3	120.4
O1-C4-C3	115.5(2)	O1-C4-C7	122.0(2)
C3-C4-C7	122.5(2)	O2-C5-O1	117.0(2)
O2-C5-C10	126.7(2)	O1-C5-C10	116.3(2)
C1-C6-C7	120.0(2)	C1-C6-H6	120.0
C7-C6-H6	120.0	C6-C7-C4	117.4(2)
C6-C7-C8	125.8(2)	C4-C7-C8	116.82(19)
C10-C8-C7	120.1(2)	C10-C8-C9	116.2(2)

C7-C8-C9	123.7(2)	O3-C9-C8	126.4(2)
O3-C9-H9	116.8	C8-C9-H9	116.8
C8-C10-C5	122.8(2)	C8-C10-H10	118.6
C5-C10-H10	118.6		

Table 7. Anisotropic atomic displacement parameters (Å²) for **2e**.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
Br1	0.0733(2)	0.0590(2)	0.03787(16)	-0.00906(12)	-0.00204(11)	0.00380(14)
O1	0.0712(11)	0.0367(9)	0.0363(9)	0.0043(7)	0.0031(8)	-0.0003(8)
O2	0.0824(13)	0.0569(13)	0.0358(9)	0.0021(9)	0.0006(8)	0.0042(10)
O3	0.140(2)	0.0400(11)	0.0475(12)	0.0087(9)	-0.0021(12)	-0.0140(12)
C1	0.0415(12)	0.0416(13)	0.0367(12)	-0.0058(10)	0.0029(9)	0.0053(11)
C2	0.0528(15)	0.0373(14)	0.0521(14)	-0.0110(11)	0.0023(11)	0.0014(11)
C3	0.0610(16)	0.0297(13)	0.0523(15)	0.0021(11)	0.0063(12)	0.0009(11)
C4	0.0469(13)	0.0346(13)	0.0349(11)	0.0004(9)	0.0049(9)	0.0060(10)
C5	0.0527(15)	0.0433(15)	0.0385(12)	0.0003(11)	0.0029(10)	0.0057(12)
C6	0.0454(13)	0.0332(12)	0.0375(12)	0.0021(10)	0.0065(9)	0.0019(10)
C7	0.0365(12)	0.0288(11)	0.0369(11)	-0.0013(9)	0.0042(9)	0.0044(9)
C8	0.0393(12)	0.0315(12)	0.0386(12)	-0.0001(9)	0.0052(9)	0.0026(10)
C9	0.0684(17)	0.0333(13)	0.0468(15)	-0.0034(11)	0.0065(12)	-0.0050(12)
C10	0.0485(14)	0.0340(12)	0.0427(13)	-0.0059(10)	0.0029(10)	-0.0003(11)

Table 8. Hydrogen atomic coordinates and isotropic atomic displacement parameters ($\text{\AA}^2$) for **2e**.

	x/a	y/b	z/c	U(eq)
H2	0.3778	0.8266	1.1025	0.057
H3	0.3224	0.8262	0.9419	0.057
H6	0.2541	0.3713	1.1076	0.046
H9	0.0941	0.0905	0.9185	0.059
H10	0.0962	0.2503	0.7941	0.05

Crystal Structure Report of compound 2h

A specimen of $C_{12}H_{12}O_3$ was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

The integration of the data using a triclinic unit cell yielded a total of 5458 reflections to a maximum θ angle of 30.54° ($0.70 \text{ \AA}$ resolution), of which 4196 were independent (average redundancy 1.301, completeness = 59.4%, $R_{\text{int}} = 3.55\%$, $R_{\text{sig}} = 10.08\%$) and 2004 (47.76%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 8.8433(3) \text{ \AA}$, $b = 9.0407(3) \text{ \AA}$, $c = 14.4291(6) \text{ \AA}$, volume = $1153.60(7) \text{ \AA}^3$, are based upon the refinement of the XYZ-centroids of reflections above $20 \sigma(I)$.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group $P -1$, with $Z = 4$ for the formula unit, $C_{12}H_{12}O_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 275 variables converged at $R1 = 19.69\%$, for the observed data and $wR2 = 51.45\%$ for all data. The goodness-of-fit was 1.551. The largest peak in the final difference

electron density synthesis was $1.712 \text{ e}^-/\text{\AA}^3$ and the largest hole was $-0.485 \text{ e}^-/\text{\AA}^3$ with an RMS deviation of $0.150 \text{ e}^-/\text{\AA}^3$. On the basis of the final model, the calculated density was 1.176 g/cm^3 and $F(000)$, 432 e^- .

Table 1. Sample and crystal data for **2h**.

Identification code	Chem_Jyoti_216	
Chemical formula	C ₁₂ H ₁₂ O ₃	
Formula weight	204.22	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	triclinic	
Space group	P -1	
Unit cell dimensions	a = 8.8433(3) Å	α = 90°
	b = 9.0407(3) Å	β = 90°
	c = 14.4291(6) Å	γ = 90°
Volume	1153.60(7) Å ³	
Z	4	
Density (calculated)	1.176 Mg/cm ³	
Absorption coefficient	0.084 mm ⁻¹	
F(000)	432	

Table 2. Data collection and structure refinement for **2h**

Theta range for data collection	2.25 to 30.54°	
Index ranges	-12<=h<=9, -11<=k<=12, -16<=l<=20	
Reflections collected	5458	
Independent reflections	4196 [R(int) = 0.0355]	
Structure solution technique	direct methods	
Structure solution program	SHELXS-97 (Sheldrick, 2008)	
Refinement method	Full-matrix least-squares on F ²	
Refinement program	SHELXL-97 (Sheldrick, 2008)	
Function minimized	$\sum w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	4196 / 0 / 275	
Goodness-of-fit on F²	1.551	
Δ/σ_{max}	1.532	
Final R indices	2004 data; I>2 σ (I)	R1 = 0.1969, wR2 = 0.4807
	all data	R1 = 0.2543, wR2 = 0.5145
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.2000P)^2+0.0000P]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	1.712 and -0.485 eÅ ⁻³	

R.M.S. deviation from mean	0.150 eÅ-3	
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Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å²) for **2h**.

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x/a	y/b	z/c	U(eq)
C7	0.8117(10)	0.0765(10)	0.0043(7)	0.065(2)
O2	0.4839(4)	0.7287(3)	0.28729(16)	0.0187(7)
O3	0.7400(8)	0.8397(7)	0.9636(4)	0.0689(17)
O4	0.6803(9)	0.6171(8)	0.9134(5)	0.089(2)
O5	0.9848(4)	0.7290(3)	0.21289(16)	0.0187(7)
O6	0.2381(8)	0.8391(7)	0.5371(4)	0.0703(18)
O7	0.1796(9)	0.6155(8)	0.5863(5)	0.088(2)
C1	0.8953(15)	0.3390(12)	0.0370(8)	0.088(3)
C2	0.8028(11)	0.2036(10)	0.0607(6)	0.061(2)

C3	0.7212(10)	0.9561(9)	0.0247(5)	0.0551(19)
C4	0.6177(10)	0.9509(10)	0.0979(5)	0.062(2)
C5	0.5256(10)	0.8193(10)	0.1059(5)	0.058(2)
C6	0.4077(12)	0.7908(10)	0.1757(6)	0.071(2)
C8	0.6549(11)	0.7147(10)	0.9697(6)	0.064(2)
C9	0.5473(11)	0.7061(9)	0.0447(6)	0.067(2)
C10	0.6104(11)	0.0833(10)	0.1527(5)	0.062(2)
C11	0.7031(12)	0.1989(9)	0.1322(5)	0.067(2)
C12	0.5124(16)	0.1043(11)	0.2360(6)	0.091(4)
C13	0.9063(11)	0.7903(10)	0.3231(6)	0.068(2)
C14	0.0289(10)	0.8210(9)	0.3949(5)	0.0575(19)
C15	0.1166(10)	0.9503(10)	0.4028(5)	0.062(2)
C16	0.2199(10)	0.9579(10)	0.4756(5)	0.0562(19)
C17	0.3096(10)	0.0778(10)	0.4946(5)	0.060(2)
C18	0.3031(10)	0.2040(9)	0.4392(6)	0.060(2)
C19	0.3960(15)	0.3386(11)	0.4630(7)	0.087(3)
C20	0.1101(11)	0.0826(10)	0.3476(5)	0.063(2)
C21	0.2018(12)	0.1993(9)	0.3671(6)	0.068(2)
C22	0.1541(12)	0.7132(10)	0.5276(6)	0.066(2)

C23	0.0477(12)	0.7056(10)	0.4581(6)	0.068(2)
C24	0.0127(16)	0.1048(11)	0.2636(6)	0.090(4)

Table 4. Bond lengths (Å) for **2h**.

C7-C3	1.383(13)	C7-C2	1.410(12)
O2-C6	1.833(9)	O3-C8	1.360(11)
O3-C3	1.383(10)	O4-C8	1.220(10)
O5-C13	1.822(9)	O6-C22	1.366(12)
O6-C16	1.403(10)	O7-C22	1.244(10)
C1-C2	1.512(14)	C1-H1A	0.96
C1-H1B	0.96	C1-H1C	0.96
C2-C11	1.358(12)	C3-C4	1.398(11)
C4-C10	1.436(12)	C4-C5	1.447(13)
C5-C9	1.365(11)	C5-C6	1.472(12)
C6-H6	0.93	C8-C9	1.443(13)
C9-H9	0.93	C10-C11	1.361(13)
C10-C12	1.494(11)	C11-H11	0.93
C12-H12A	0.9604	C12-H12B	0.9604

C12-H12C	0.9604	C13-C14	1.525(12)
C13-H13	0.93	C14-C23	1.395(11)
C14-C15	1.407(13)	C15-C16	1.393(11)
C15-C20	1.438(12)	C16-C17	1.371(12)
C17-C18	1.395(11)	C17-H17	0.93
C18-C21	1.374(12)	C18-C19	1.509(14)
C19-H19A	0.96	C19-H19B	0.96
C19-H19C	0.96	C20-C21	1.361(13)
C20-C24	1.501(12)	C21-H21	0.93
C22-C23	1.377(13)	C23-H23	0.93
C24-H24A	0.96	C24-H24B	0.96
C24-H24C	0.96		

Table 5. Bond angles (°) for **2h**.

C3-C7-C2	119.1(8)	C8-O3-C3	121.6(7)
C22-O6-C16	120.8(7)	C2-C1-H1A	109.3
C2-C1-H1B	109.7	H1A-C1-H1B	109.5
C2-C1-H1C	109.4	H1A-C1-H1C	109.5

H1B-C1-H1C	109.5	C11-C2-C7	116.7(9)
C11-C2-C1	123.2(9)	C7-C2-C1	119.9(9)
O3-C3-C7	113.2(7)	O3-C3-C4	122.3(8)
C7-C3-C4	124.5(8)	C3-C4-C10	114.7(9)
C3-C4-C5	117.1(8)	C10-C4-C5	128.1(8)
C9-C5-C4	119.1(8)	C9-C5-C6	114.3(8)
C4-C5-C6	126.6(8)	C5-C6-O2	113.2(7)
C5-C6-H6	123.5	O2-C6-H6	123.3
O4-C8-O3	117.1(9)	O4-C8-C9	125.6(9)
O3-C8-C9	117.2(7)	C5-C9-C8	122.5(9)
C5-C9-H9	118.6	C8-C9-H9	118.9
C11-C10-C4	119.6(8)	C11-C10-C12	115.3(8)
C4-C10-C12	125.1(9)	C10-C11-C2	125.4(8)
C10-C11-H11	117.2	C2-C11-H11	117.4
C10-C12-H12A	109.3	C10-C12-H12B	109.8
H12A-C12-H12B	109.4	C10-C12-H12C	109.4
H12A-C12-H12C	109.4	H12B-C12-H12C	109.4
C14-C13-O5	112.2(6)	C14-C13-H13	124.0
O5-C13-H13	123.8	C23-C14-C15	120.2(8)

C23-C14-C13	113.1(8)	C15-C14-C13	126.7(8)
C16-C15-C14	117.6(8)	C16-C15-C20	113.7(9)
C14-C15-C20	128.6(8)	C17-C16-C15	124.7(8)
C17-C16-O6	114.3(7)	C15-C16-O6	121.0(9)
C16-C17-C18	120.5(8)	C16-C17-H17	119.7
C18-C17-H17	119.8	C21-C18-C17	115.9(9)
C21-C18-C19	123.5(9)	C17-C18-C19	120.5(8)
C18-C19-H19A	109.1	C18-C19-H19B	109.2
H19A-C19-H19B	109.5	C18-C19-H19C	110.1
H19A-C19-H19C	109.5	H19B-C19-H19C	109.5
C21-C20-C15	120.5(8)	C21-C20-C24	113.9(8)
C15-C20-C24	125.6(9)	C20-C21-C18	124.7(8)
C20-C21-H21	117.7	C18-C21-H21	117.6
O7-C22-C23	125.8(10)	O7-C22-O6	115.1(9)
C23-C22-O6	119.1(8)	C22-C23-C14	121.3(9)
C22-C23-H23	119.3	C14-C23-H23	119.4
C20-C24-H24A	109.4	C20-C24-H24B	109.4
H24A-C24-H24B	109.5	C20-C24-H24C	109.7
H24A-C24-H24C	109.5	H24B-C24-H24C	109.5

Table 6. Anisotropic atomic displacement parameters (Å²) for **2h**.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
C7	0.045(5)	0.066(5)	0.084(6)	-0.004(5)	-0.002(5)	-0.002(4)
O2	0.0249(18)	0.0226(14)	0.0087(11)	0.0040(9)	-0.0042(10)	-0.0025(12)
O3	0.067(4)	0.077(4)	0.063(3)	-0.013(3)	0.011(3)	0.000(3)
O4	0.090(6)	0.080(4)	0.098(5)	-0.035(4)	0.007(4)	0.002(4)
O5	0.0265(18)	0.0219(14)	0.0078(11)	-0.0043(9)	0.0035(10)	-0.0030(12)
O6	0.067(4)	0.076(4)	0.068(3)	0.014(3)	-0.008(3)	0.004(4)
O7	0.085(5)	0.083(4)	0.096(5)	0.041(4)	-0.007(4)	0.002(4)
C1	0.083(8)	0.083(7)	0.096(7)	0.009(6)	-0.024(6)	0.000(7)
C2	0.060(6)	0.061(4)	0.063(4)	0.004(4)	-0.009(4)	-0.001(4)
C3	0.053(5)	0.065(4)	0.047(3)	0.003(3)	-0.002(3)	0.011(4)
C4	0.051(5)	0.083(5)	0.051(3)	0.006(3)	0.002(3)	0.013(4)

C5	0.058(5)	0.066(5)	0.050(4)	0.004(3)	-0.006(3)	0.000(4)
C6	0.059(6)	0.085(5)	0.068(5)	0.003(4)	-0.009(4)	0.001(5)
C8	0.060(6)	0.064(5)	0.067(5)	-0.008(4)	-0.013(4)	0.001(4)
C9	0.076(6)	0.057(4)	0.068(5)	-0.001(4)	-0.009(4)	-0.006(4)
C10	0.066(6)	0.070(5)	0.050(4)	0.000(3)	0.005(4)	0.007(4)
C11	0.088(7)	0.056(4)	0.057(4)	-0.002(3)	0.000(4)	0.000(5)
C12	0.128(10)	0.070(6)	0.075(6)	0.000(5)	0.035(6)	0.012(7)
C13	0.056(5)	0.082(5)	0.067(5)	-0.006(4)	-0.001(4)	-0.004(5)
C14	0.059(5)	0.067(5)	0.046(3)	-0.004(3)	0.009(3)	0.000(4)
C15	0.047(5)	0.083(5)	0.056(4)	-0.004(4)	-0.007(3)	0.016(4)
C16	0.057(5)	0.067(5)	0.044(3)	0.001(3)	0.006(3)	0.012(4)
C17	0.048(5)	0.074(5)	0.059(4)	0.005(4)	-0.005(3)	0.002(4)
C18	0.057(5)	0.060(4)	0.063(4)	-0.005(3)	0.009(4)	0.003(4)
C19	0.092(8)	0.073(6)	0.095(7)	0.007(5)	0.025(6)	-0.003(6)
C20	0.070(6)	0.071(5)	0.050(4)	0.000(3)	0.003(4)	0.011(5)
C21	0.083(7)	0.056(4)	0.065(4)	0.002(4)	0.002(4)	0.003(5)
C22	0.070(6)	0.066(5)	0.062(4)	0.013(4)	0.008(4)	0.007(5)
C23	0.078(7)	0.057(4)	0.069(5)	0.004(4)	0.016(5)	-0.005(5)
C24	0.124(11)	0.070(5)	0.077(6)	0.012(5)	-0.035(6)	0.000(7)

Table 7. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å²) for **2h**.

	x/a	y/b	z/c	U(eq)
H1A	0.8862	0.4105	0.0859	0.131
H1B	0.8591	0.3811	-0.0199	0.131
H1C	0.9995	0.3115	0.0300	0.131
H6	0.3050	-0.1969	0.1644	0.085
H9	0.4906	-0.3798	0.0516	0.08
H11	0.6978	0.2816	0.1704	0.08
H12A	0.5309	0.2002	0.2623	0.136
H12B	0.5353	0.0294	0.2811	0.136
H12C	0.4081	0.0968	0.2183	0.136
H13	-0.1966	-0.1984	0.3343	0.082
H17	0.3753	0.0749	0.5450	0.072
H19A	0.3378	0.4263	0.4513	0.13
H19B	0.4858	0.3401	0.4256	0.13
H19C	0.4236	0.3353	0.5273	0.13
H21	0.1954	0.2819	0.3289	0.081
H23	-0.0128	-0.3782	0.4531	0.082
H24A	-0.0915	0.1095	0.2820	0.135

H24B	0.0268	0.0237	0.2215	0.135
H24C	0.0404	0.1955	0.2335	0.135