Supporting Information

A chemodosimeter for ratiometric detection of cyanide in aqueous media and human blood serum

Sandeep Kumar, Prabhpreet Singh, Geeta Hundal, Maninder Singh Hundal and Subodh Kumar*

Department of Chemistry, UGC Centre for Advanced Studies, Guru Nanak Dev University,

Amritsar, 143 005, India.

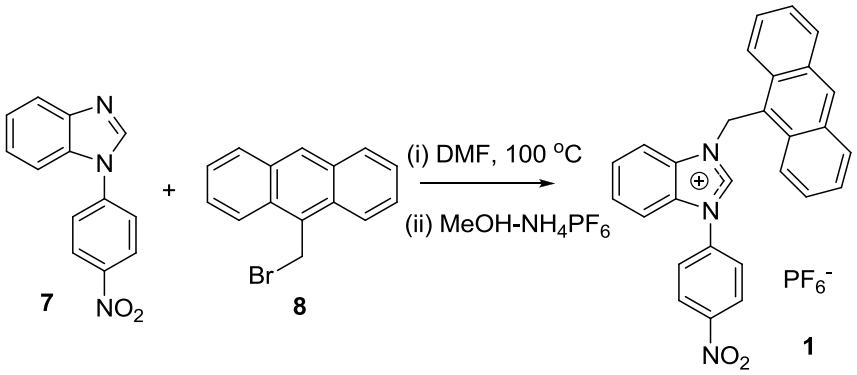
Mobile: +91 9872361528. E-mail address: subodh_gndu@yahoo.com

Content:

1. Experimental Section:

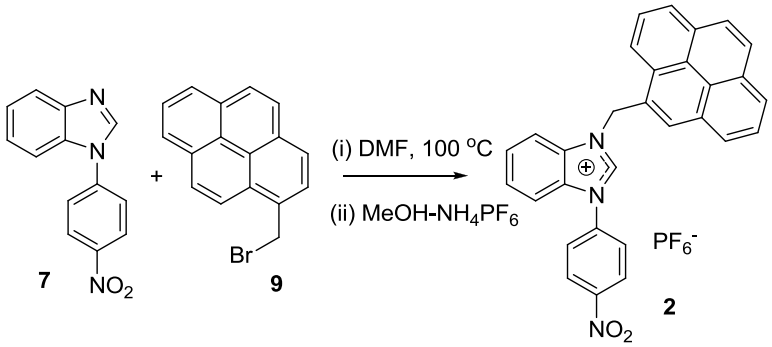
General experimental conditions: All reagents were purchased from commercial suppliers and used without further purification. Solvents used were purified and dried by standard methods prior to use. TLC analyses were performed on silica gel plates and column chromatography was conducted over silica gel (mesh 100–200). ^{1}H NMR spectra and titrations were carried out using JEOL A1 spectrometer operating at 300 MHz at GNDU, Amritsar or using Brucker 400 MHz machine at RSIC, Chandigarh. ^{13}C NMR sepctra were recorded at 75 MHz. All chemical shifts are reported in ppm relative to the TMS as an internal reference. UV-Vis studies were carried out on a Shimadzu UV-1601 PC or Shimadzu UV-2400 machines using slit width of 1.0 nm and matched quartz cells. HRMS spectra were recorded on Brucker MicroToff/QII. The fluorescence experiments were performed on Shimadzu 1501 fluorescence spectrophotometer.

Synthesis of 1.



9-(Bromomethyl)anthracene (1 mmol, 275 mg) **8** and 1-(4-nitrophenyl)benzimidazole **7** (1.5 mmol, 358.5 mg) were dissolved in DMF (5 ml) and heated at 100 °C for 24 h. The solvent was removed under reduced pressure. Crude solid thus obtained was dissolved in methanol (10 ml). To this methanolic solution of compound was added aqueous NH_4PF_6 (1 mmol, 163 mg) dropwise with continuous stirring for 3-4 hr. The solid separated was filtered, dried and crystallized from CH_3CN-IPA (isopropyl alcohol) (8:2) mixture to get yellow colored crystals of **1**. Yield 69%; M. Pt. 205 ^oC; ^1H NMR (DMSO-d_6): δ 6.80 (s, 2H, CH_2), 7.63 (t, 4H, *J* = 7.5 Hz, ArH), 7.82 (d, 1H, *J* = 7.2 Hz, ArH), 7.89 (d, 4H, *J* = 8.1 Hz, ArH), 8.25 (d, 2H, *J* = 7.5 Hz, ArH), 8.42 (d, 5H, *J* = 8.4 Hz, ArH), 8.91 (s, 1H, ArH), 9.57 (s, 1H, BimC2H). ^{13}C NMR (DMSO-d_6): δ ^{13}C NMR (100 MHz, DMSO) δ 45.3, 132.7, 134.1, 142.2, 145.3, 147.2, 147.8, 150.0, 150.5, 150.8, 152.6, 154.2, 154.8, 154.9, 155.1, 155.6, 163.0, 168.3, 175.9. HRMS *m/z* (TOF MS ES^+) calculated for $C_{28}H_{20}N_3O_2$ [M^+] 430.1550; found 430.1545; Found C, 58.48; H, 3.65; N, 7.49%; $C_{28}H_{20}N_3O_2PF_6$ requires C, 58.44; H, 3.50; N, 7.30 %).

Synthesis of 2.



chemodosimeter 2. Yield 78%; M. pt. 235 °C; ^{1}H NMR (DMSO-d_6): δ 6.68 (s, 2H, CH_2), 7.77 (t, 2H, *J* = 4.0 Hz, ArH), 7.97 (t, 1H, *J* = 4.8 Hz, ArH), 8.14-8.43 (m, 11H, ArH), 8.58 (d, 2H, *J* = 8.8 Hz, ArH), 8.65 (d, 1H, *J* = 8.0 Hz, ArH), 10.35 (s, 1H, Bim C2H). ^{13}C NMR (DMSO-d_6): δ 48.7, 113.7, 114.5, 125.1, 125.5, 125.7, 125.8, 126.9, 128.7, 128.8, 131.2, 131.4, 138.1, 143.3, 148.1. HRMS *m/z* (TOF MS ES$^+$) calculated for $C_{30}H_{20}N_3O_2$ [M$^+$] 454.1550; found 454.1551; Found C, 60.55; H, 3.79; N, 7.19%; $C_{30}H_{20}N_3O_2PF_6$ requires C, 60.11; H, 3.36; N, 7.01 %).

2.UV-Vis and fluorescence behavior of Chemodosimeter 1, 2 and formamide 3

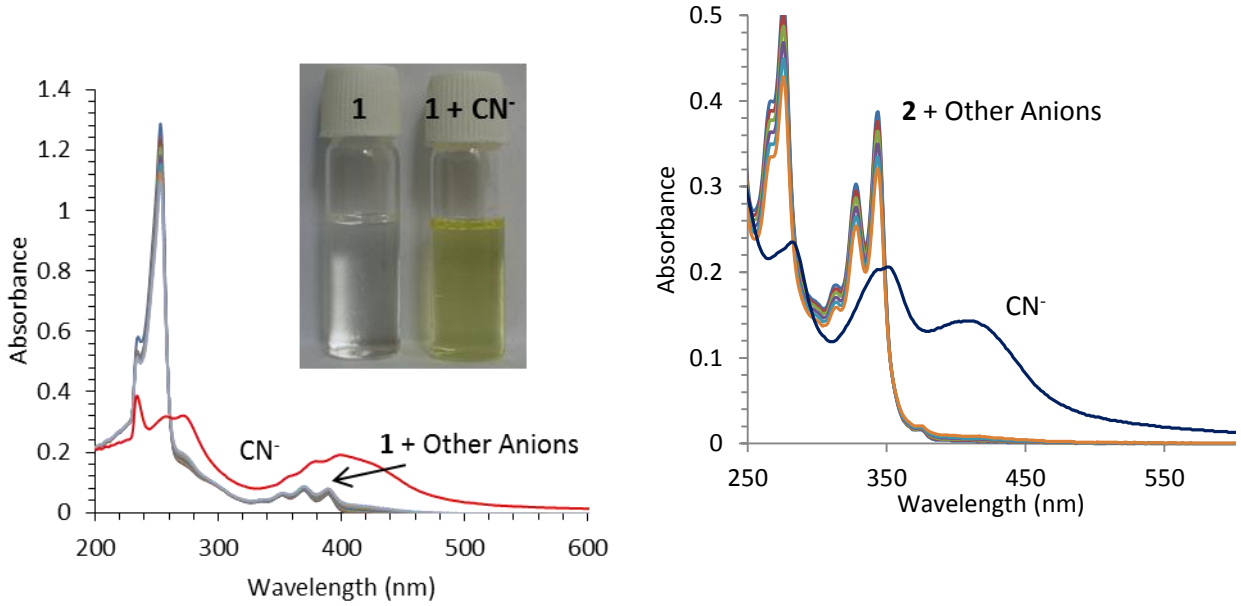


Figure S1. Effect of different anions as sodium/potassium salts on the UV-Vis spectrum of **1** (a); and **2** (b); 10 µM in HEPES buffered solution at pH 7.4.

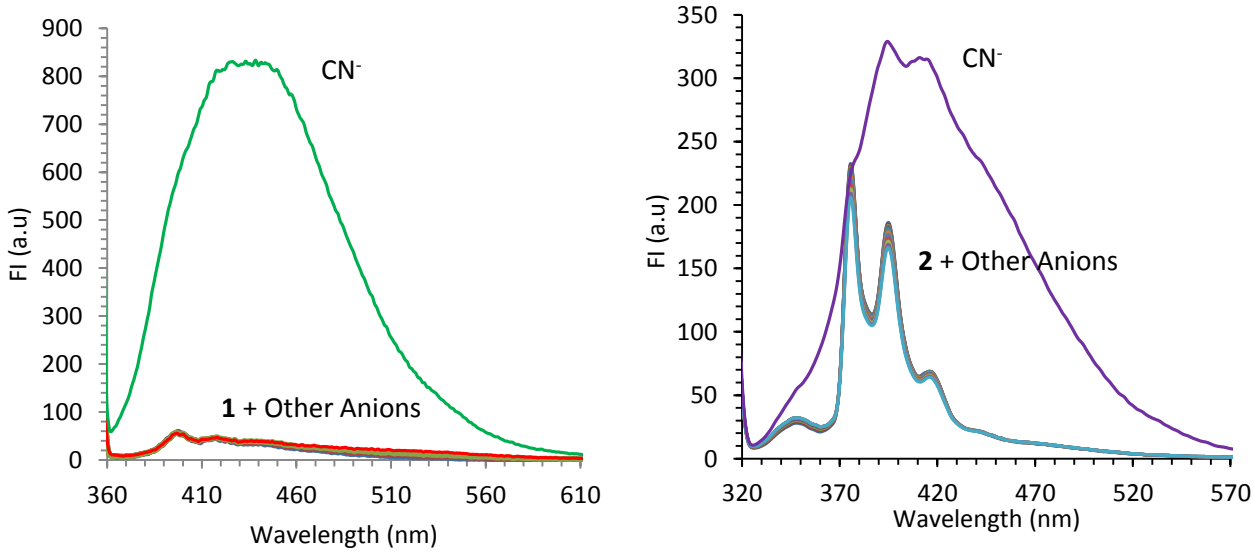


Figure S2. Effect of different anions as sodium/potassium salts on the fluorescence spectrum of **1** (a); and **2** (b); 10 µM in HEPES buffered solution at pH 7.4.

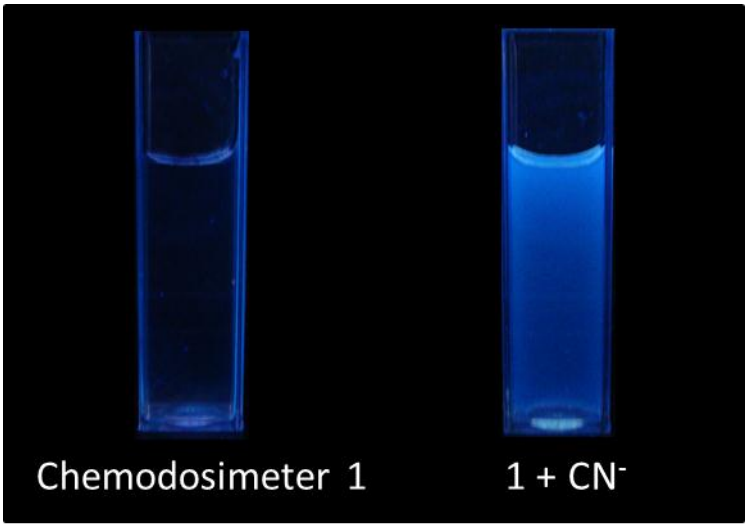


Figure S3. Fluorescence images captured under UV-lamp on irradiation at 365 nm before and after addition of 10 equivalents of cyanide ions to HEPES solution (5% DMSO) of chemodosimeter **1** (10 µM).

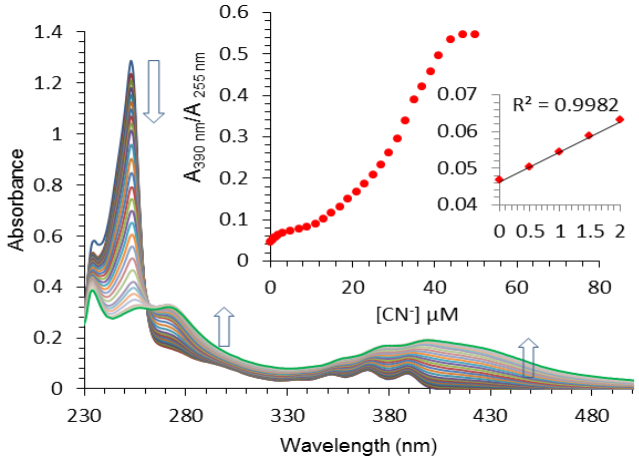


Figure S4. Effect of gradual addition of NaCN on the UV-Vis spectrum of **1** (10 µM, HEPES-DMSO; 19:1, pH 7.4). Inset: Plot of $A_{390\ nm}$ / $A_{255\ nm}$ Vs [CN^-].

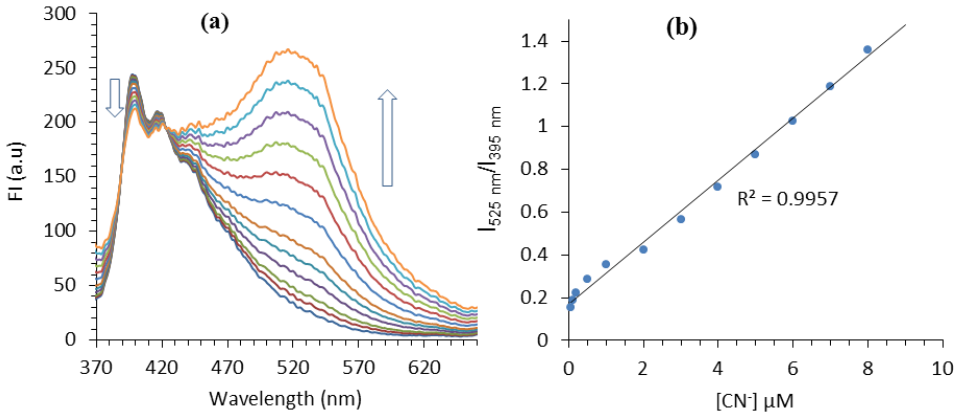


Figure S5. (a) Effect of gradual addition of NaCN on the fluorescence intensity of **1** (10 µM, HEPES buffer-DMSO; 19:1, pH 7.4, λ_{ex} 350 nm, slit width: ex 5, em 10); (b) ratiometric plot of $FI_{525\ nm}/FI_{395\ nm}$ vs [CN^-].

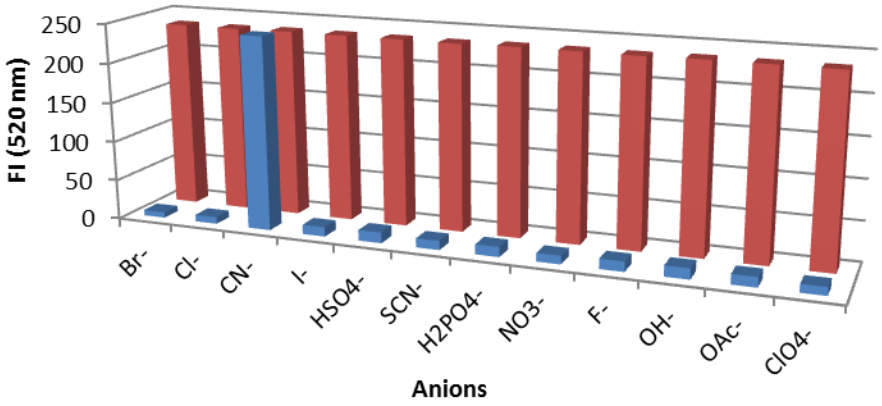


Figure S6. Fluorescence response of **1** (10 µM) to various anions in HEPES-DMSO (19:1) pH 7.4; λ_{ex} = 350 nm. Blue bars represent selectivity of **1** upon addition of different anions; red bars represent competitive selectivity of probe **1** towards CN^- ions (10 µM) in the presence of other anions (100 µM).

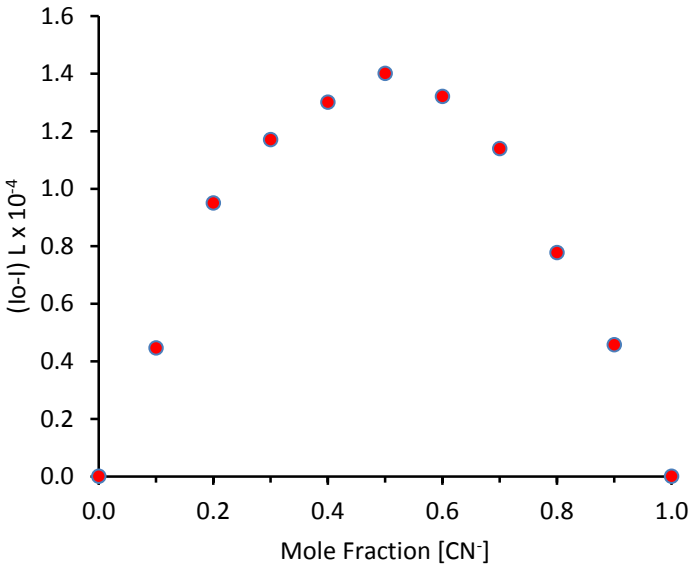


Figure S7. Job's Plot analysis of **1** with NaCN determined by emission channel in HEPES buffer (95% aqueous), pH 7.4.

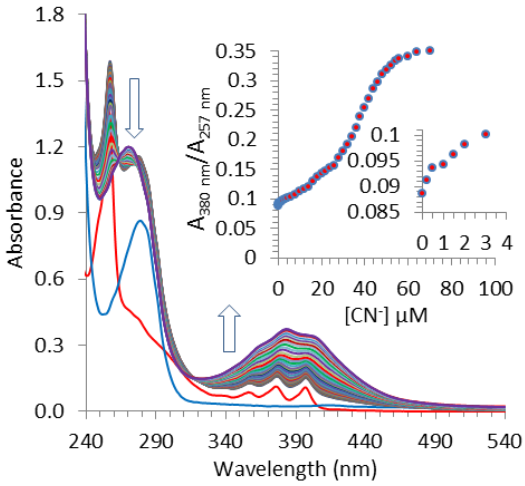


Figure S8a. Effect of addition of NaCN on the UV-Vis spectrum of probe **1** (10 µM, Blood Serum) in HEPES buffered solution, pH 7.4, (blue trace shows the absorption spectrum of blood serum, red trace shows the absorption spectrum of probe **1**). Inset showing the plot of $A_{380\ nm}$ / $A_{257\ nm}$ vs [CN^-] on addition of NaCN solution with lowest detection limit of 0.25 µM.

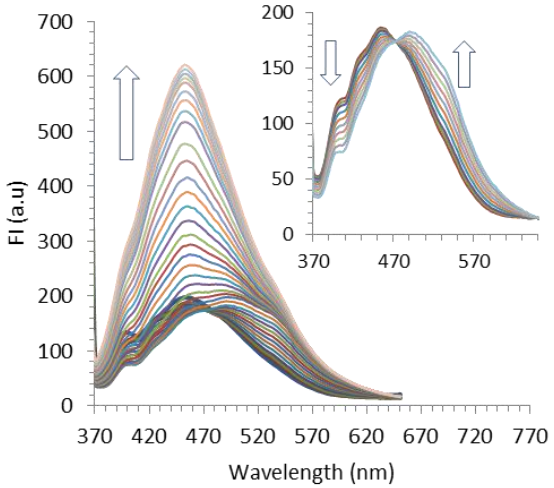


Figure S8b. Effect of gradual addition of NaCN on emission spectrum of probe **1** (10 µM, blood serum) in HEPES buffered solution, pH 7.4. λ_{ex} 350 nm.

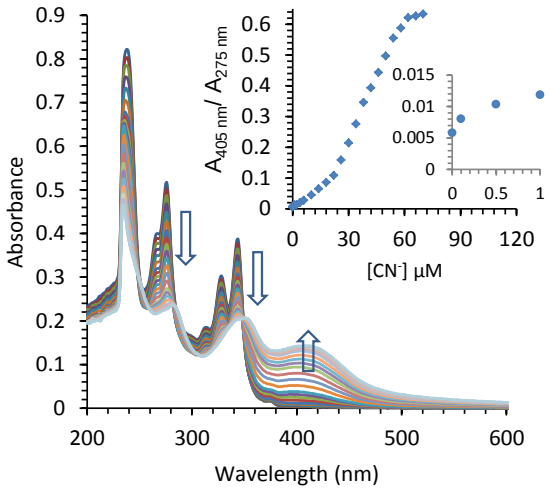


Figure S9a. Effect of gradual addition of NaCN on UV-Vis spectrum of **2** (10 µM) in HEPES buffered solution, pH 7.4. The binding constants determined using SPECFIT-32 model were found to be log $\beta_{L(CN)}$ = 6.15 ± 0.37 and log$\beta_{L(CN)2}$ = 10.06 ± 0.36 for 1:1 and 1:2 (3-CN) stoichiometries.

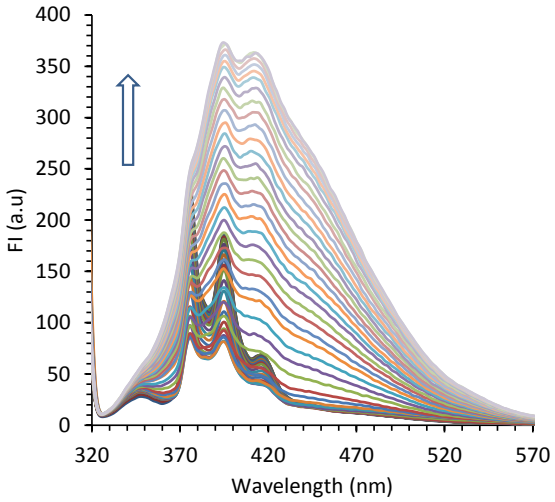
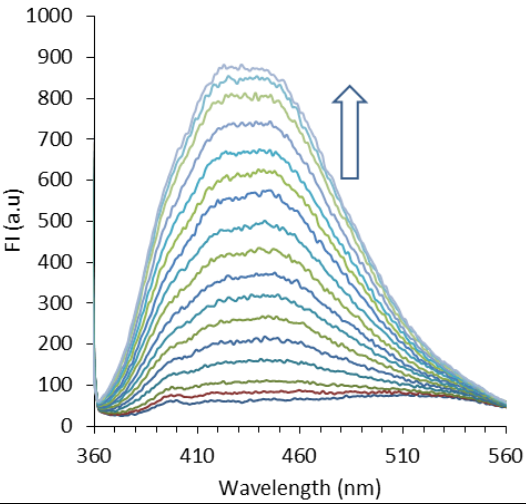


Figure S9b. Effect of gradual addition of NaCN on fluorescence spectrum of **2** (10 µM) in HEPES buffered solution, pH 7.4. The binding constants determined using SPECFIT-32 model were found to be log$\beta_{L(CN)3}$ = 13.98 ± 0.07 for 1:3 (**2**-CN) stoichiometries. (λ_{ex} 350 nm). Again by fitting the graph separately for decrease in emission intensity gave the binding constant value log$\beta_{L(CN)2}$ = 10.43 ± 0.31 for 1:2 (**2**-CN) stoichiometry and for increase in emission intensity gave log$\beta_{L(CN)2}$ = 9.25 ± 0.25 for 1:2 (**2**-CN) stoichiometry.



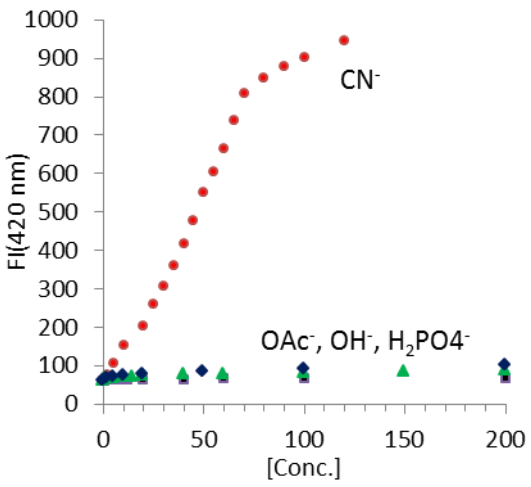


Figure S10. (a) Effect of gradual addition of NaCN on the emission spectrum of **3** (10 µM) in HEPES-DMSO (19:1), pH 7.4; (b) Concentration vs. FI plot of various anions viz. CN^-, OH^-, OAc^- and $H_2PO_4^-$ showing no change except CN^- in emission intensity in 1 µM-100 µM range.

Table 1: Comparison of the different sensors and their detection limit published in the literature

	Published	Solvent	Detection limits	Serum studies	Organic molecule	Ratiometric*
1	New J. Chem., 2010, 34, 132–136	water–ACN 1 : 1	10 μM at 50 ^{o}C for 55 min.	No	Ir complex	No
2	Adv. Funct. Mater. 2010, 20, 951–956	Water	200 nM	No	Au NCs	No
3	*Chem. Commun.*, 2010, **46**, 7951–7953	water	10 µM	No	yes	Yes-C
4	Tetrahedron Letters 51 (2010) 5810–5814	Water	20 µM	No	Yes	Yes-C
5	Tetrahedron Letters 51 (2010) 4712–4716	PBS buffer	10 µM	No	Cu-ensemble	Yes-C
6	*Chem. Commun.*, 2010, **46**, 8953–8955	HEPES	5 µM	No	Yes	Yes-C
7	Chemical Physics Letters 484 (2010) 168–172	Water	2 µM	No	Yes	Yes-C
8	Org. Lett. 2010, 3604	ACN:Buffer (1:1)	60 nM	No	Yes	Yes-F
9	Tetrahedron 66 (2010) 1678–1683	HEPES	2.48 ppm	No	Cu-ensemble	Yes-F
10	Tetrahedron 66 (2010) 1846–1851	MeOH:Buffer		No	Cu-ensemble	Yes-C
11	Anal. Chimica Acta, 2010, 657, 69-74	Water	16 ug L^{-1}	No	Yes	No
12	Intern. J Environ. Anal. Chem., 2010, 90, 148-158.	Water	20 ngL^{-1}	No	Co-centre	Yes-C
13	Tetrahedron 67 (2011) 4196-4201	Water	0.33 µM	No	Yes	Yes-C
14	Tetrahedron 67 (2011) 4939-4947	Water	11 nM polymer	No	Yes	Yes-C
15	Sensors and Actuators B., 2011, 157, 26-33	Water	10 µM	No	Yes	Yes-C
16	ACS Appl. Mater. Intrfaces., 2011, 3, 4649-4656.	CH_3CN-H_2O (2:8)	0.5 µM	No	Yes	No
17	Anal. Chim. Acta, 2011, 694, 120	Water	1.29 µM	No	Yes	Yes-C
18	Anal. Chem. 2011, 83, 2712–2718	Water	18 ug/L	No	Cu-ensemble	No
19	Chem Commun., 2011, 47, 3805-3807.	Water		No	Cu-ensemble	No
20						
21	Tetrahedron, 2011, 67, 5678-5685	Water	270 nM	No	Yes	Yes-C
23	Chem. Eur. J. 2011, 17, 2057 – 2062.	MethaNol-H_2O , HEPES (6:4)	1 µM or 26 ppb	No	Yes	No
24	OL 2011, 5056	HEPES	100 nM 20 min.	No	Cu-ensemble	No
25	Small, 2012, 8, 612-618.	Water	100 nM	No	Yes	No
26	*Chem. Commun.*, 2012, **48**, 2707–	ACN-Buffer	2 µM	No	Ir Complex	No

	2709	1:1				
27	Chem. Eur. J. 2012, 18, 4513-4516	Water		No	Yes	No
28	Chem. Asian. J. 2012, 7, 2805-2812.	99% H2O	0.38 ppm	No	Yes	No
29	Analyst, 2012, 137, 5581-5585	EtOH-H_2O (4:6, NaH_2PO_4/Na_3PO_4 buffer, 20 mM, pH 9.4)	0.4 μM	No	Yes	Yes-C
30	Tet. Lett. 2012, 53, 5455-5457	HEPES:DMSO (1:1)	5.3 μM	No	Yes	Yes-F
31	CC, 2012, **48**, 3942–3944.	Aqueous solution	80 nM Solid support	Yes	Mesoporous graphitic carbon(IV) nitride	No
32	Tetrahedron 68 (2012) 2523-2526	water/THF 40:60, v/v)	6.6 μM (12 h)	No	Yes	Yes-C
33	Talanta *89 (2012) 264– 269*.	HEPES	4.03 μM	No	Cu-ensemble	No
34	Sensors and Actuators B *161 (2012) 510– 519*.	EtOH-H_2O (3:7)	1 μM	No	Yes	Yes-C
35	Sensors and Actuators B *168 (2012) 14– 19*	HEPES buffer	0.5 μM,	No	Cu-ensemble	No
36	Chem. Commun., 2012, 48, 12195-12197.	Water	91 nM AIEE	No	Yes	No
37	Sensors and Actuator B: Chemicals, 2012, 168, 365-369	Water	10 μM	No	Yes	
38	Dalton Trans., 2012, 41, 11413-11418.	Water/serum		Yes	Cu-ensemble	No
39	Analyst, 2012, 137, 2333-2337.	Water	2 μM	No	Zn-ensemble	No
	Present manuscript	HEPES (5% DMSO)	30 nM (0.08 ppb)	Yes	Yes	Yes

3. NMR Spectral analysis of 1 and 2 (^{1}H, ^{13}C NMR)

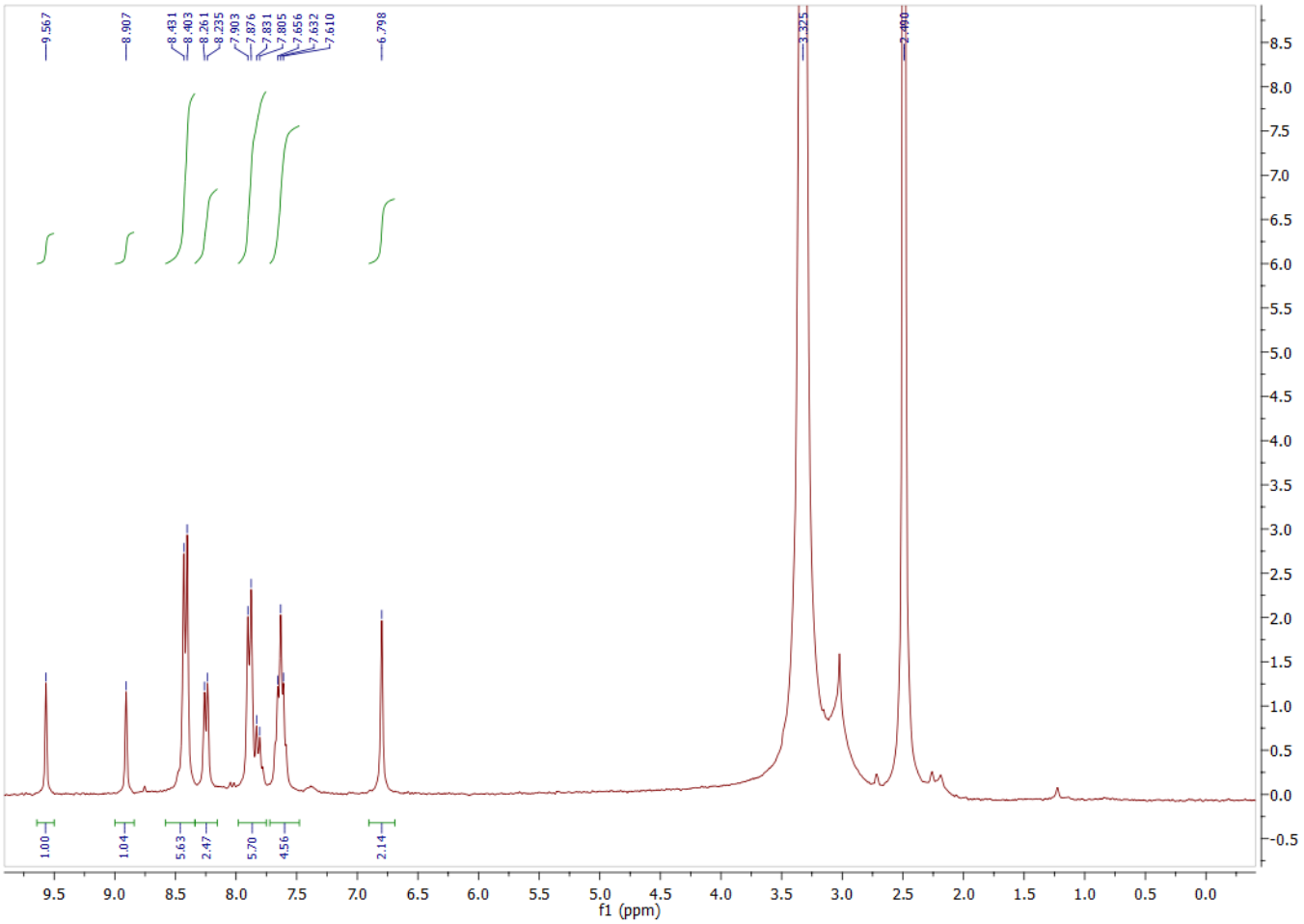


Figure S11. ^{1}H NMR spectrum of **1** in DMSO-d_6.

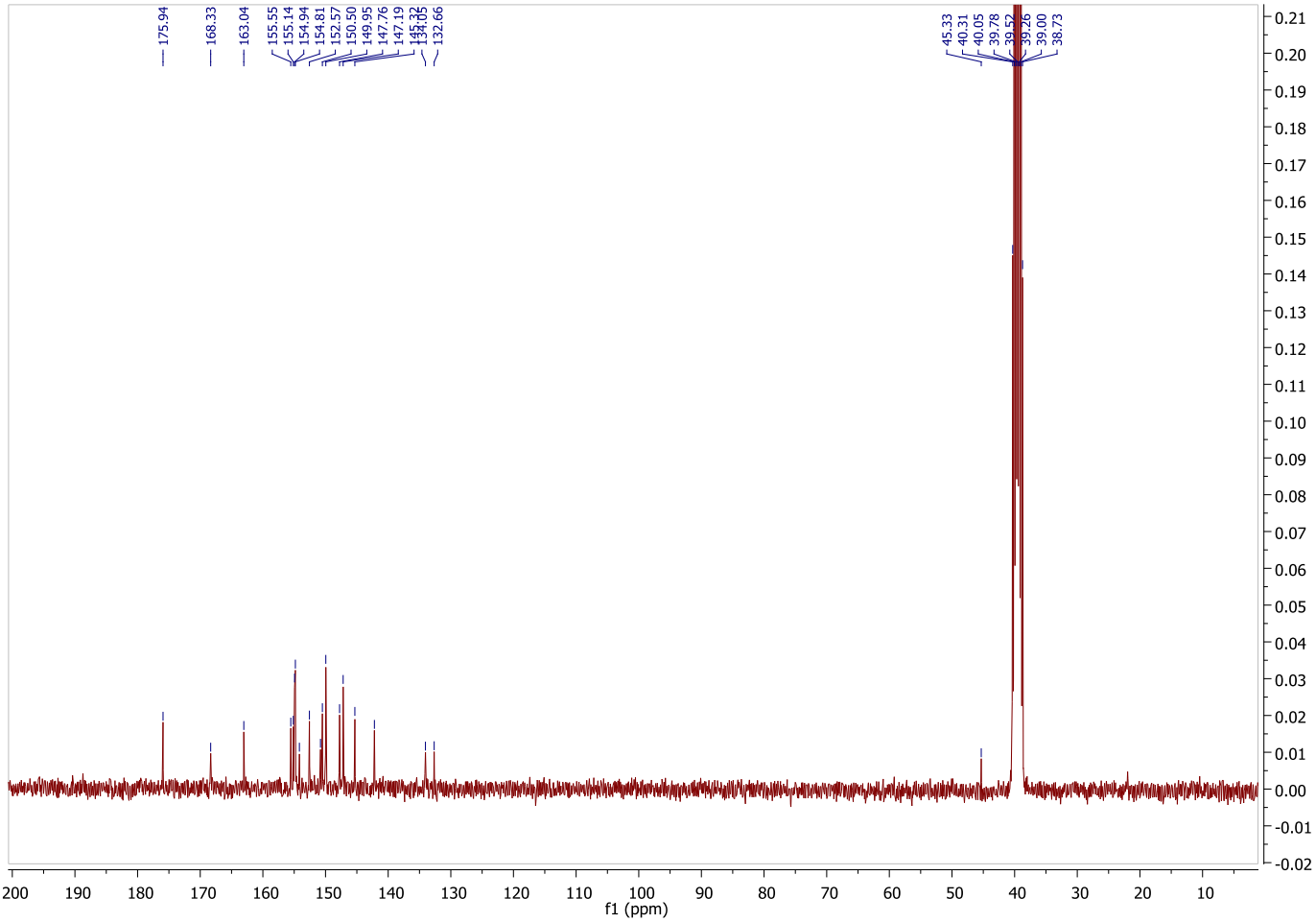


Figure S12. ^{13}C NMR of **1** in DMSO-d_6.

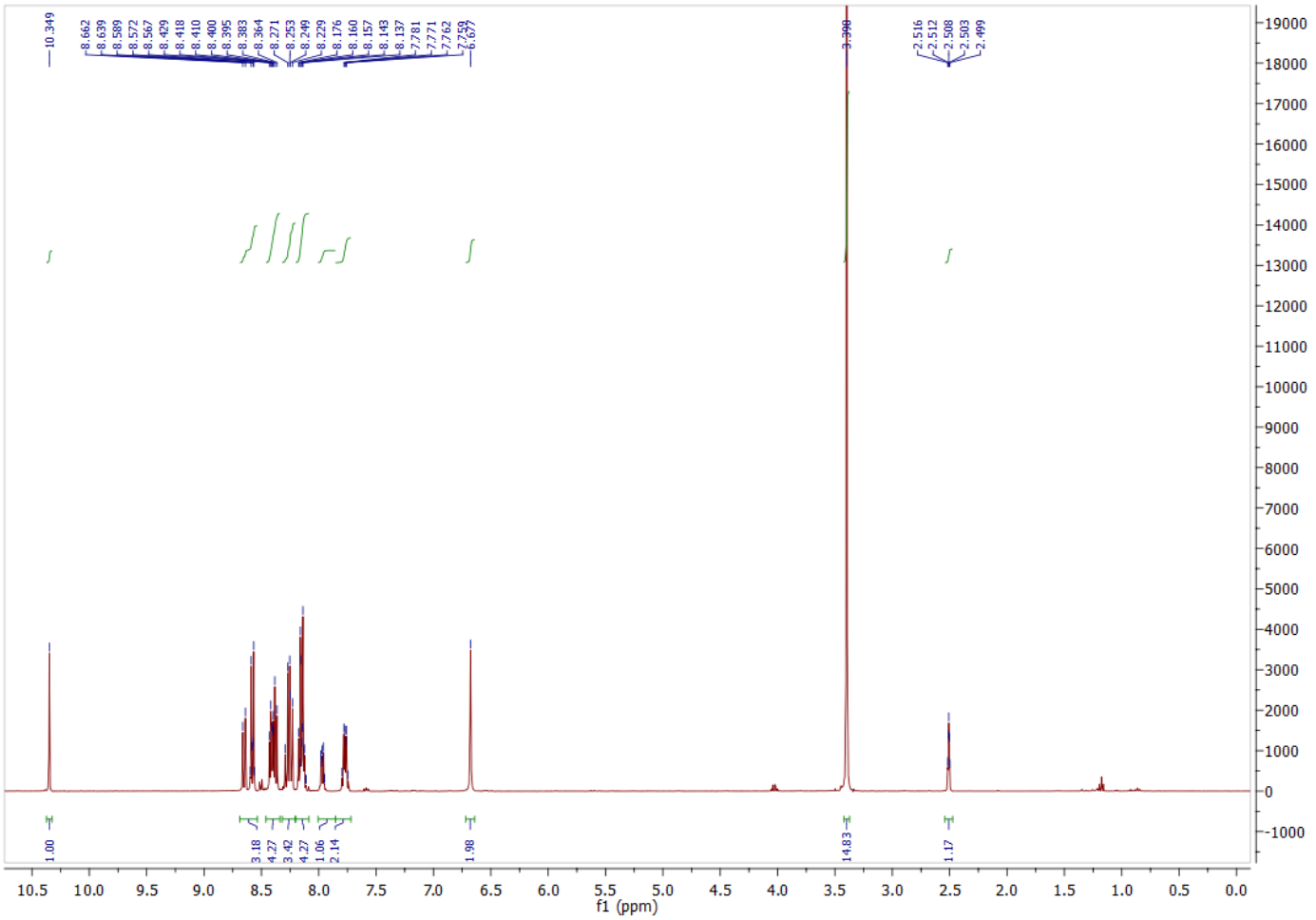


Figure S13. ^{1}H NMR of **2** in DMSO-d_6.

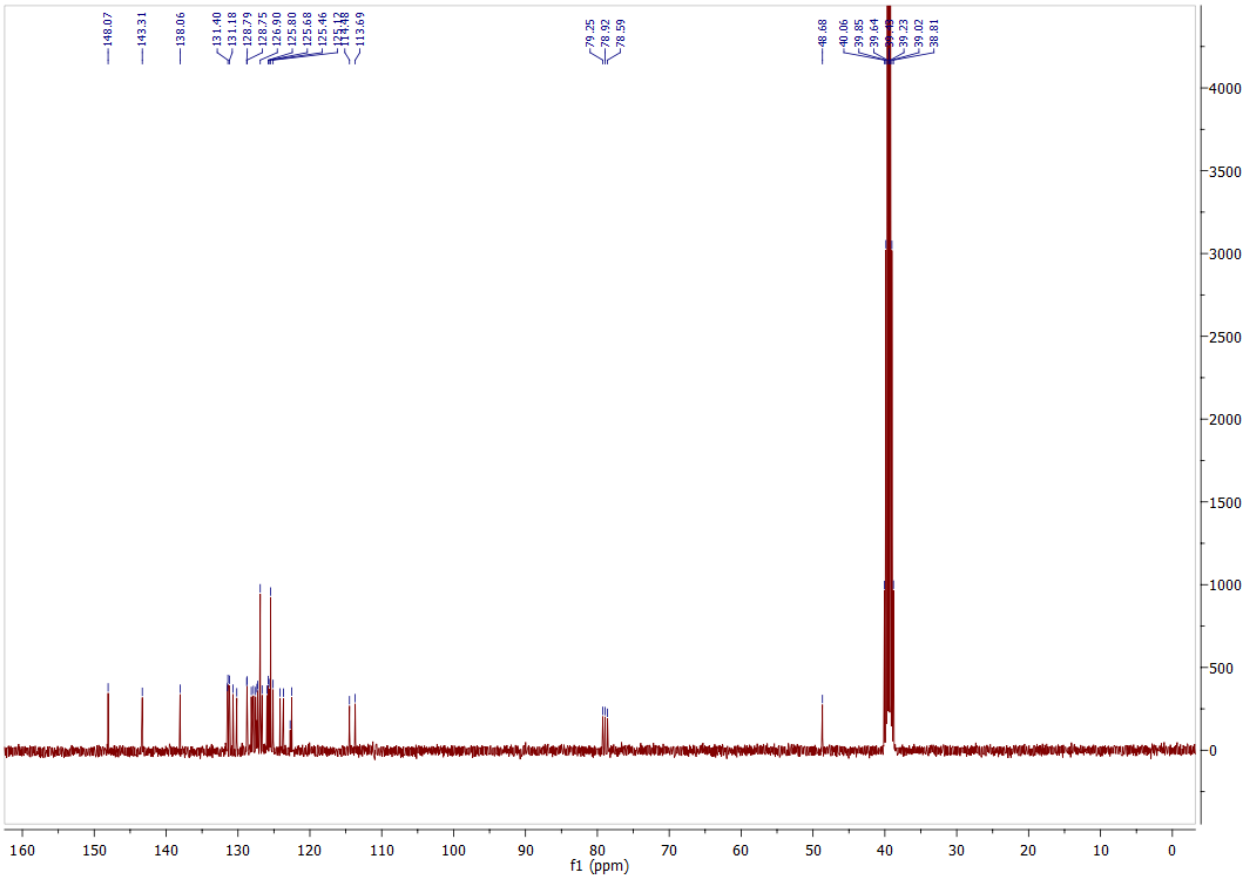


Figure S14. ^{13}C NMR of **2** in DMSO-d_6.

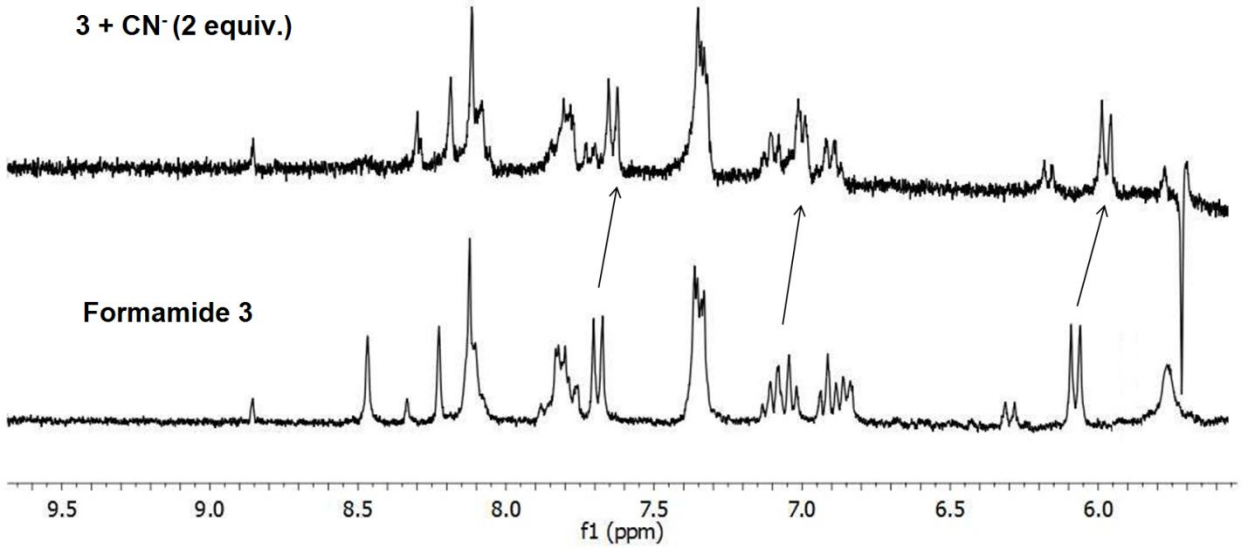


Figure S15. Partial NMR spectra of formamide **3** before and after addition of 2 equivalent of CN^- ions.

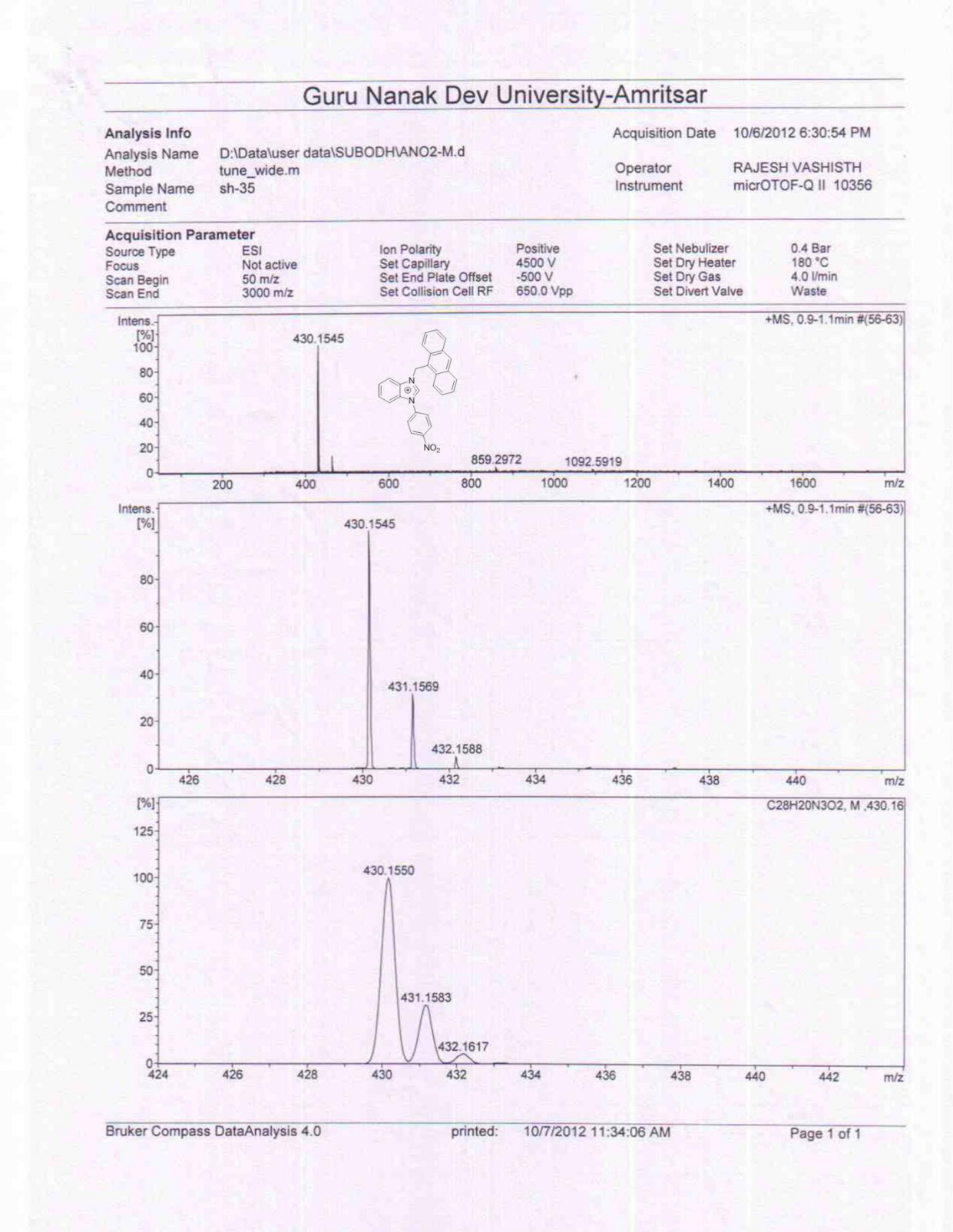


Figure S16. HRMS spectrum of **1**.

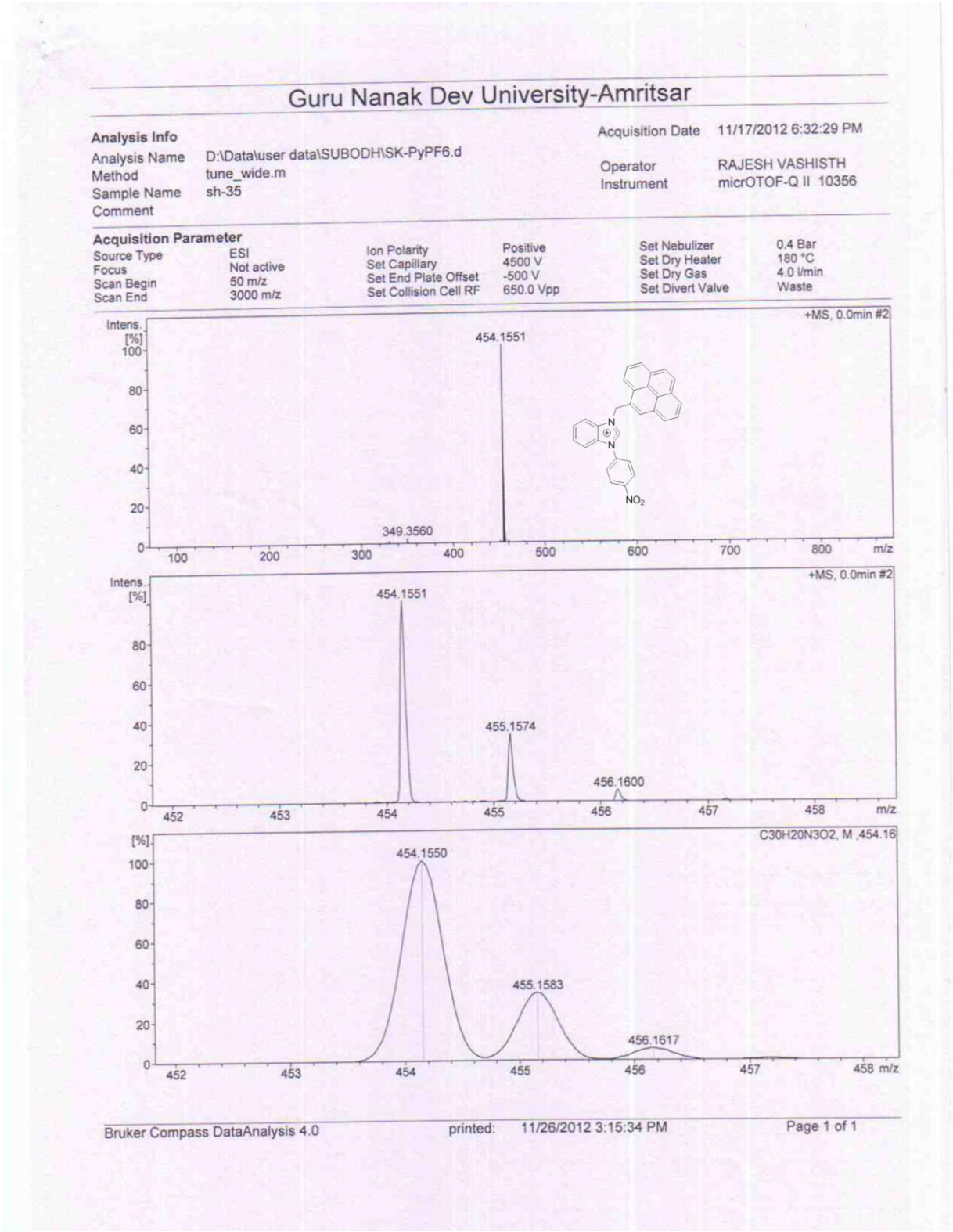


Figure S17. HRMS spectrum of **2**.

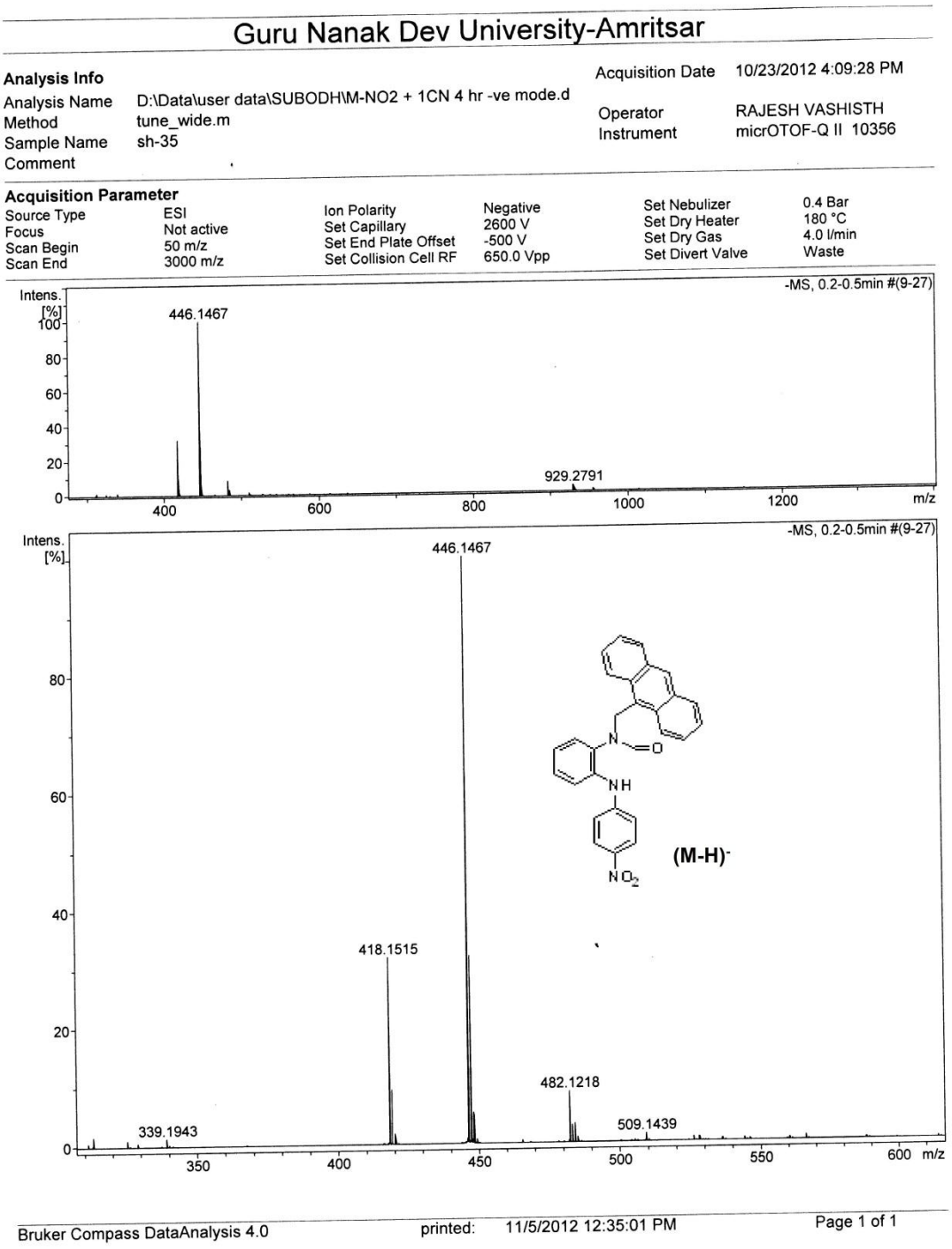


Figure S18. HRMS spectrum of formamide **3** showing $[M - H]^-$ ion peak.

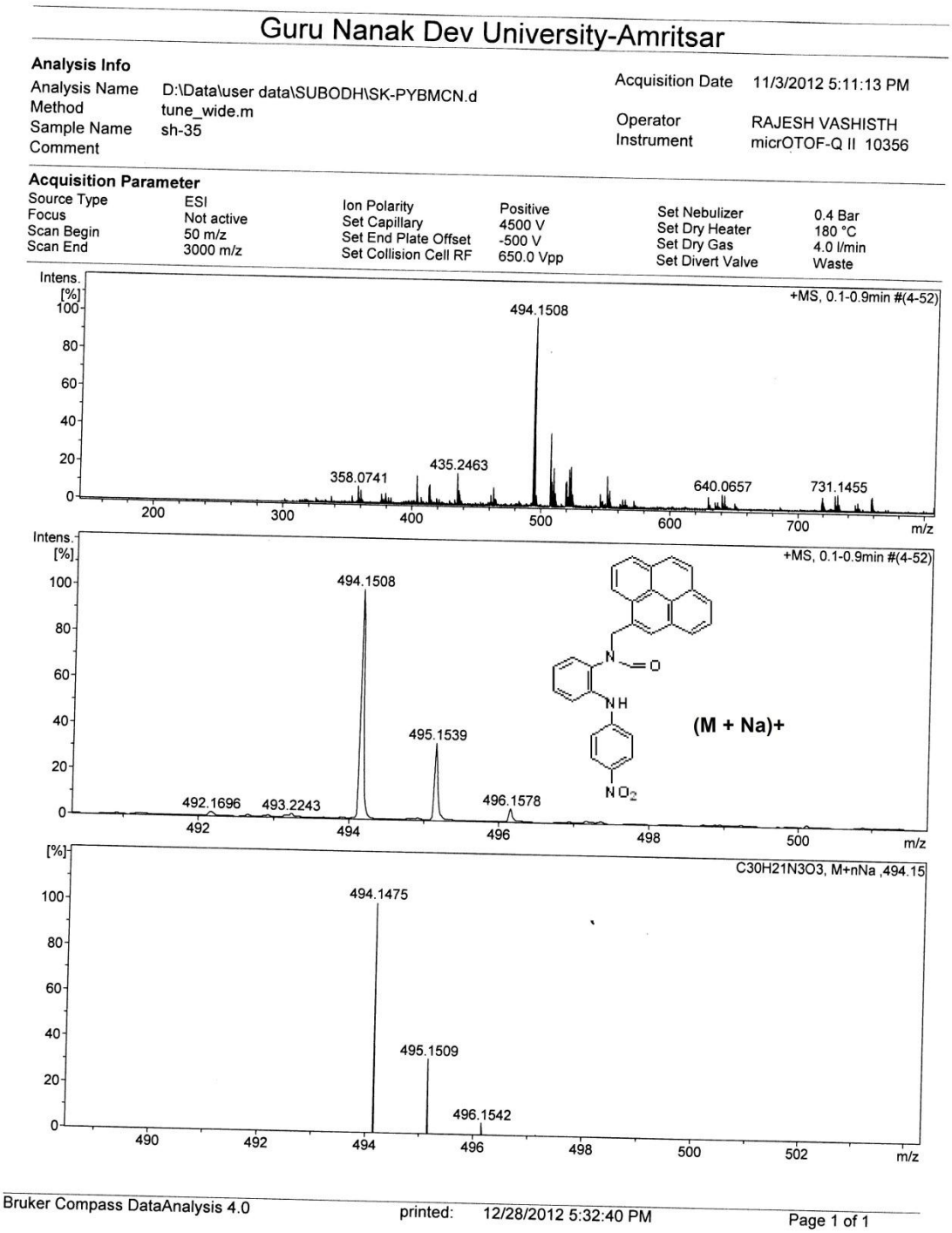


Figure S19. HRMS spectrum of formamide **5** showing [M + Na^+] ion peak.

Detection limit The detection limit was calculated based on the fluorescence titration. To determine the S/N ratio, the emission intensity of **1** (10 μM) without cyanide was measured by 5 times and the standard deviation of blank measurements was determined. Under the present conditions, a good linear relationship between the fluorescence intensity and the cyanide concentration could be obtained in the 0 - 10 μM (R^2 = 0.9957). The detection limit is then calculated with the equation:

$$\text{Detection limit} = 3\sigma bi/m$$

Where, σbi is the standard deviation of blank measurements; m is the slope between intensity versus sample concentration. The detection limit was measured to be 30 nM at S/N = 3.

Computational Studies:

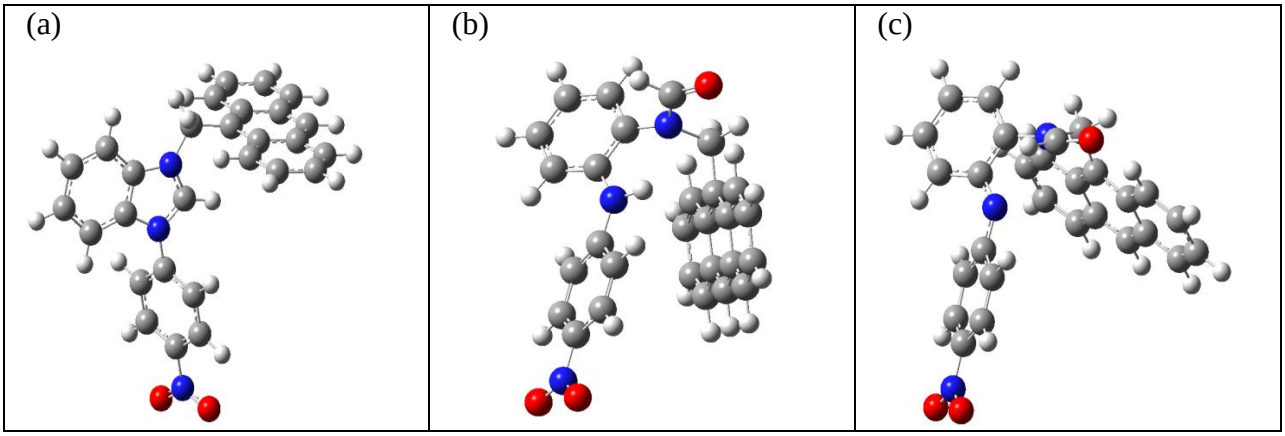


Figure S20. Geometrically optimized structures at B3LYP-321G (a) Probe **1**; (b) Formamide derivative **3**; (c) deprotonated form of formamide **3**.

5. X-ray structure data

Experimental

X-ray data of all three compounds was collected on a Bruker's Apex-II CCD diffractometer using Mo Kα (λ=0.71069). The data were corrected for Lorentz and polarization effects and empirical absorption corrections were applied using SADABS from Bruker. For the chemodosimeter **1** a total of 30726 reflections were measured out of which 7910 were independent and 2545 were observed [I>2 σ(I)] for theta 30.60°, for the product (**3**) a total of 17503 reflections were measured out of which 5008 were independent and 2623 were observed [I>2 σ(I)] for theta 27.45°,for the product (**5**) a total of 17751 reflections were measured out of which 5828 were independent and 3120 were observed [I>2 σ(I)] for theta 27.26°. Crystals of compounds (**1**) and (**3**) were of poor quality. The ratio of observed/unique reflections for (**1**) was only 32% which did not improve on measuring the data twice on different crystals. The data for (**3**) were measured three times on different crystals from three different lots. The given results are from the best data set. All three structures were solved by direct methods using SIR-92 [1] and refined by full-matrix least squares refinement methods based on F^2, using SHELX-97 [2]. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were fixed geometrically with their U_{iso} values 1.2 times of the phenylene and methylene carbons. All calculations were performed using Wingx [3] package. Important crystal and refinement parameters are given in **Table 1**. All non-hydrogen atoms in all three structures have been refined anisotropically. (**1**) has a PF_6^- counter anion which shows the expected disorder in terms of high thermal parameters of the F atoms. No attempts were made to resolve this disorder in wake of the less observed data. Compound (**3**) has disorder in its terminal anthracene group which showed high thermal parameters and anomalous bond distances for C-C bonds. All attempts to resolve this disorder failed, therefore the group was refined with restraints on its bond lengths. The disorder is reflected in the cif causing some A and B alerts. However both the structures do not show any significant residual electron density and all the bond lengths are angles (except the disordered anthracene ring in (**3**)) are normal. The crystal structure of (**1**) shows solvent accessible voids of 32 $Å^3$ which are due to crystal packing forces. The structure of (**5**) has a solvent DMSO molecule in it which is disordered. The refinement shows small peaks <0.45 Å near S and O of the solvent but this disorder could not be resolved. The disorder manifests itself in terms of the non-planarity of the molecule with all the bond lengths and thermal parameters being normal. The nitro group of the nitrophenyl ring also showed disorder in one of the oxygen atoms (O2) that could be successfully resolved by splitting it at two atomic positions with 60 and 40% occupancy.

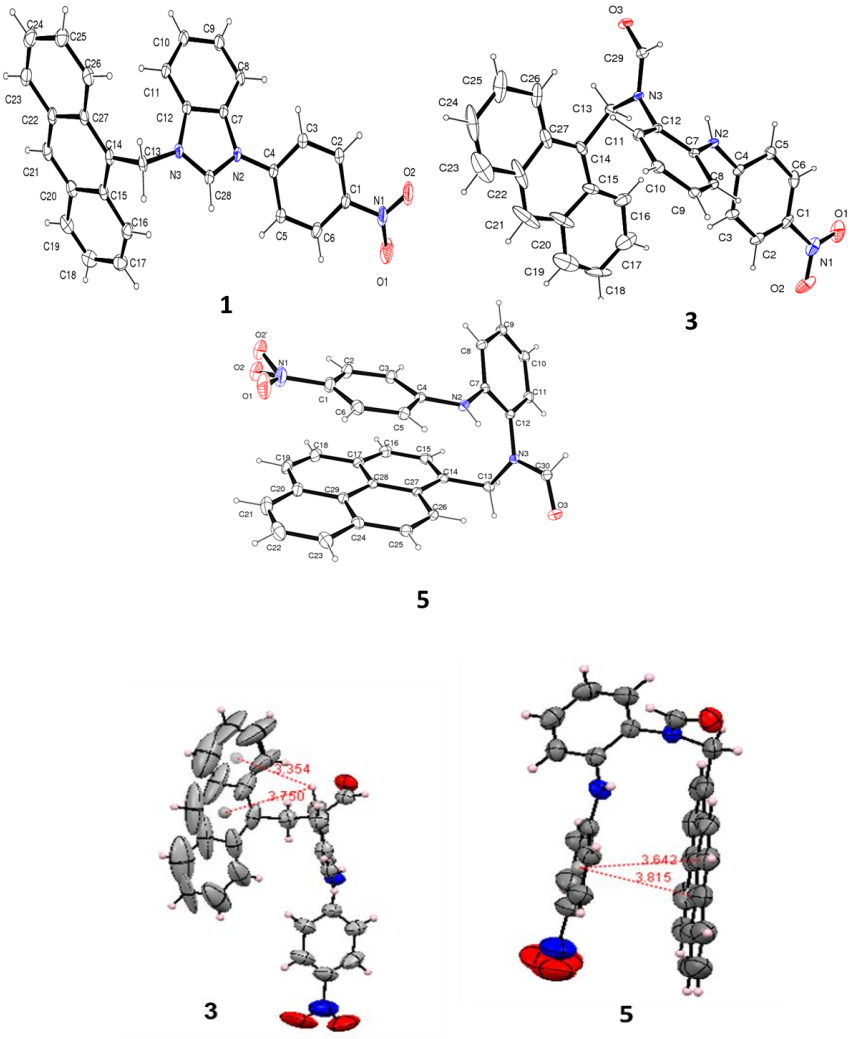


Figure S21. ORTEP diagram (left) of the chemodosimeter (**1**). Anion PF_6^- has been removed for clarity, (right) of the formamide (**3**) (below) of formamide **5**, The solvent has been removed. The disorder in the nitro group is shown.

[1] A. Altomare, G. Cascarano, C. Giacovazzo and A. Guagliardi, *J. Appl. Crystallogr.* **1993**, 26, 343-350.

[2] G. M. Sheldrick, *Acta Cryst A*, **2008**, A64, 112-122.

[3] L. J. Farrugia, *J. Appl. Cryst.*, **1999**, 32, 837-838.

Chemodosimeter 1

Table 2-1. Crystal data and structure refinement for Chemodosimeter **1** (sk_ano2bm_m_0m)

CCDC no. 914969

Identification code	shelxl	
Empirical formula	C28 H20 F6 N3 O2 P	
Formula weight	575.44	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 22.5075(12) Å	□= 90°.
	b = 12.6461(7) Å	□= 118.996(3)°.
	c = 20.6804(11) Å	□ = 90°.
Volume	5148.5(5) $Å^3$	
Z	8	
Density (calculated)	1.485 Mg/m^3	
Absorption coefficient	0.183 mm^{-1}	
F(000)	2352	
Crystal size	0.13 x 0.11 x 0.09 mm^3	
Theta range for data collection	1.91 to 30.60°.	
Index ranges	-32<=h<=32, -18<=k<=11, -29<=l<=29	
Reflections collected	30726	
Independent reflections	7910 [R(int) = 0.0902]	
Completeness to theta = 30.60°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7461 and 0.6328	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7910 / 1 / 349	
Goodness-of-fit on F^2	0.998	
Final R indices [I>2sigma(I)]	R1 = 0.0963, wR2 = 0.2562	
R indices (all data)	R1 = 0.2625, wR2 = 0.3421	
Largest diff. peak and hole	0.819 and -0.488 $e.Å^{-3}$	

Table 2-2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)

For chemodosimeter **1** sk_ano2bm_m_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	6792(2)	2808(2)	2754(2)	50(1)
C(2)	6456(2)	3362(2)	3061(2)	63(1)
C(3)	6271(2)	4410(2)	2865(2)	60(1)
C(4)	6421(2)	4904(2)	2361(2)	45(1)
C(5)	6757(2)	4350(2)	2054(2)	53(1)
C(6)	6942(2)	3302(2)	2250(2)	56(1)
C(7)	6478(2)	6790(3)	2700(2)	48(1)
C(8)	6909(3)	6796(4)	3456(2)	65(1)
C(9)	7057(3)	7756(4)	3795(3)	78(2)
C(10)	6778(3)	8678(4)	3416(3)	72(2)
C(11)	6347(3)	8686(4)	2665(2)	59(1)
C(12)	6204(2)	7725(3)	2314(2)	46(1)
C(13)	5347(2)	8052(3)	920(2)	50(1)
C(14)	5641(2)	9107(3)	873(2)	45(1)
C(15)	6118(2)	9117(4)	616(2)	47(1)
C(16)	6368(2)	8186(4)	451(3)	59(1)
C(17)	6819(3)	8221(5)	194(3)	74(2)
C(18)	7040(3)	9194(6)	61(3)	80(2)
C(19)	6825(3)	10093(5)	204(3)	72(2)
C(20)	6360(2)	10112(4)	495(2)	56(1)
C(21)	6123(2)	11044(4)	640(2)	58(1)
C(22)	5670(2)	11044(4)	908(2)	56(1)
C(23)	5435(3)	12015(4)	1058(3)	70(2)
C(24)	4995(3)	12029(5)	1325(3)	79(2)
C(25)	4752(3)	11087(5)	1459(3)	78(2)
C(26)	4949(2)	10157(3)	1323(2)	66(1)
C(27)	5416(2)	10068(4)	1037(2)	49(1)

C(28)	5865(2)	6379(3)	1522(2)	46(1)
N(1)	6976(3)	1740(4)	2950(3)	78(1)
N(2)	6258(2)	5969(2)	2191(2)	45(1)
N(3)	5816(2)	7411(3)	1565(2)	44(1)
O(1)	7333(3)	1314(4)	2751(3)	139(2)
O(2)	6766(3)	1296(3)	3309(3)	121(2)
F(1)	4662(3)	4663(5)	553(5)	226(4)
F(2)	3649(4)	4641(4)	-409(2)	181(3)
F(3)	3789(2)	4326(4)	666(2)	140(2)
F(4)	3351(2)	5790(4)	173(4)	203(3)
F(5)	4290(4)	6087(4)	10(3)	182(2)
F(6)	4364(3)	5850(5)	1055(3)	197(3)
P(1)	3997(1)	5258(1)	312(1)	66(1)

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Table 2-3. Bond lengths [Å] and angles [°] for chemodosimeter **1** sk_ano2bm_m_0m.

C(1)-C(2)	1.3900
C(1)-C(6)	1.3900
C(1)-N(1)	1.413(5)
C(2)-C(3)	1.3900
C(2)-H(2)	0.9300
C(3)-C(4)	1.3900
C(3)-H(3)	0.9300
C(4)-C(5)	1.3900
C(4)-N(2)	1.395(4)
C(5)-C(6)	1.3900
C(5)-H(5)	0.9300
C(6)-H(6)	0.9300
C(7)-C(8)	1.385(6)
C(7)-N(2)	1.387(5)
C(7)-C(12)	1.392(6)
C(8)-C(9)	1.361(7)
C(8)-H(8)	0.9300
C(9)-C(10)	1.374(7)
C(9)-H(9)	0.9300
C(10)-C(11)	1.375(6)
C(10)-H(10)	0.9300
C(11)-C(12)	1.372(6)
C(11)-H(11)	0.9300
C(12)-N(3)	1.417(5)
C(13)-N(3)	1.478(5)
C(13)-C(14)	1.512(6)
C(13)-H(13A)	0.9700
C(13)-H(13B)	0.9700
C(14)-C(15)	1.410(6)
C(14)-C(27)	1.420(6)
C(15)-C(16)	1.416(6)
C(15)-C(20)	1.441(6)
C(16)-C(17)	1.356(7)
C(16)-H(16)	0.9300

C(17)-C(18)	1.403(8)
C(17)-H(17)	0.9300
C(18)-C(19)	1.323(8)
C(18)-H(18)	0.9300
C(19)-C(20)	1.438(7)
C(19)-H(19)	0.9300
C(20)-C(21)	1.385(7)
C(21)-C(22)	1.374(7)
C(21)-H(21)	0.9300
C(22)-C(23)	1.430(7)
C(22)-C(27)	1.439(6)
C(23)-C(24)	1.347(8)
C(23)-H(23)	0.9300
C(24)-C(25)	1.394(8)
C(24)-H(24)	0.9300
C(25)-C(26)	1.335(6)
C(25)-H(25)	0.9300
C(26)-C(27)	1.439(6)
C(26)-H(26)	0.9300
C(28)-N(3)	1.317(5)
C(28)-N(2)	1.334(5)
C(28)-H(28)	0.9300
N(1)-O(1)	1.194(6)
N(1)-O(2)	1.197(6)
F(1)-P(1)	1.527(5)
F(2)-P(1)	1.521(4)
F(3)-P(1)	1.573(4)
F(4)-P(1)	1.497(4)
F(5)-P(1)	1.526(4)
F(6)-P(1)	1.540(4)

C(2)-C(1)-C(6)	120.0
C(2)-C(1)-N(1)	120.0(3)
C(6)-C(1)-N(1)	120.0(3)
C(3)-C(2)-C(1)	120.0
C(3)-C(2)-H(2)	120.0

C(1)-C(2)-H(2)	120.0
C(2)-C(3)-C(4)	120.0
C(2)-C(3)-H(3)	120.0
C(4)-C(3)-H(3)	120.0
C(5)-C(4)-C(3)	120.0
C(5)-C(4)-N(2)	120.5(2)
C(3)-C(4)-N(2)	119.5(2)
C(6)-C(5)-C(4)	120.0
C(6)-C(5)-H(5)	120.0
C(4)-C(5)-H(5)	120.0
C(5)-C(6)-C(1)	120.0
C(5)-C(6)-H(6)	120.0
C(1)-C(6)-H(6)	120.0
C(8)-C(7)-N(2)	131.1(4)
C(8)-C(7)-C(12)	121.1(4)
N(2)-C(7)-C(12)	107.7(4)
C(9)-C(8)-C(7)	116.7(4)
C(9)-C(8)-H(8)	121.7
C(7)-C(8)-H(8)	121.7
C(8)-C(9)-C(10)	122.2(5)
C(8)-C(9)-H(9)	118.9
C(10)-C(9)-H(9)	118.9
C(11)-C(10)-C(9)	121.8(5)
C(11)-C(10)-H(10)	119.1
C(9)-C(10)-H(10)	119.1
C(12)-C(11)-C(10)	116.6(4)
C(12)-C(11)-H(11)	121.7
C(10)-C(11)-H(11)	121.7
C(11)-C(12)-C(7)	121.6(4)
C(11)-C(12)-N(3)	133.6(4)
C(7)-C(12)-N(3)	104.8(4)
N(3)-C(13)-C(14)	113.5(3)
N(3)-C(13)-H(13A)	108.9
C(14)-C(13)-H(13A)	108.9
N(3)-C(13)-H(13B)	108.9
C(14)-C(13)-H(13B)	108.9

H(13A)-C(13)-H(13B)	107.7
C(15)-C(14)-C(27)	120.4(4)
C(15)-C(14)-C(13)	118.0(4)
C(27)-C(14)-C(13)	121.5(4)
C(14)-C(15)-C(16)	123.1(4)
C(14)-C(15)-C(20)	119.7(4)
C(16)-C(15)-C(20)	117.2(4)
C(17)-C(16)-C(15)	121.8(5)
C(17)-C(16)-H(16)	119.1
C(15)-C(16)-H(16)	119.1
C(16)-C(17)-C(18)	120.6(6)
C(16)-C(17)-H(17)	119.7
C(18)-C(17)-H(17)	119.7
C(19)-C(18)-C(17)	120.4(5)
C(19)-C(18)-H(18)	119.8
C(17)-C(18)-H(18)	119.8
C(18)-C(19)-C(20)	121.8(5)
C(18)-C(19)-H(19)	119.1
C(20)-C(19)-H(19)	119.1
C(21)-C(20)-C(19)	122.8(5)
C(21)-C(20)-C(15)	119.1(4)
C(19)-C(20)-C(15)	118.1(5)
C(22)-C(21)-C(20)	121.8(4)
C(22)-C(21)-H(21)	119.1
C(20)-C(21)-H(21)	119.1
C(21)-C(22)-C(23)	120.8(5)
C(21)-C(22)-C(27)	120.8(5)
C(23)-C(22)-C(27)	118.3(5)
C(24)-C(23)-C(22)	121.6(5)
C(24)-C(23)-H(23)	119.2
C(22)-C(23)-H(23)	119.2
C(23)-C(24)-C(25)	120.6(6)
C(23)-C(24)-H(24)	119.7
C(25)-C(24)-H(24)	119.7
C(26)-C(25)-C(24)	120.5(6)
C(26)-C(25)-H(25)	119.8

C(24)-C(25)-H(25)	119.8
C(25)-C(26)-C(27)	122.7(5)
C(25)-C(26)-H(26)	118.7
C(27)-C(26)-H(26)	118.7
C(14)-C(27)-C(26)	125.5(4)
C(14)-C(27)-C(22)	118.1(4)
C(26)-C(27)-C(22)	116.4(4)
N(3)-C(28)-N(2)	110.4(4)
N(3)-C(28)-H(28)	124.8
N(2)-C(28)-H(28)	124.8
O(1)-N(1)-O(2)	122.5(5)
O(1)-N(1)-C(1)	119.0(5)
O(2)-N(1)-C(1)	118.5(5)
C(28)-N(2)-C(7)	108.1(3)
C(28)-N(2)-C(4)	126.7(3)
C(7)-N(2)-C(4)	125.3(3)
C(28)-N(3)-C(12)	109.0(3)
C(28)-N(3)-C(13)	121.9(3)
C(12)-N(3)-C(13)	128.6(3)
F(4)-P(1)-F(2)	92.9(4)
F(4)-P(1)-F(1)	172.2(5)
F(2)-P(1)-F(1)	91.4(4)
F(4)-P(1)-F(5)	100.7(4)
F(2)-P(1)-F(5)	94.0(3)
F(1)-P(1)-F(5)	85.5(4)
F(4)-P(1)-F(6)	88.6(4)
F(2)-P(1)-F(6)	178.1(4)
F(1)-P(1)-F(6)	86.9(4)
F(5)-P(1)-F(6)	86.8(3)
F(4)-P(1)-F(3)	86.9(3)
F(2)-P(1)-F(3)	86.8(3)
F(1)-P(1)-F(3)	86.8(3)
F(5)-P(1)-F(3)	172.3(3)
F(6)-P(1)-F(3)	92.2(3)

Symmetry transformations used to generate equivalent atoms:

Formamide 3 (with anthracene)

Table 6-1. Crystal data and structure refinement for formamide **3** sk_ano2bm_cn_0m. **CCDC no. 914970**

Identification code	shelxl	
Empirical formula	C28 H21 N3 O3	
Formula weight	447.48	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 8.718(3) Å	□= 65.498(16)°.
	b = 12.036(4) Å	□= 86.432(17)°.
	c = 12.296(4) Å	□ = 78.223(15)°.
Volume	1149.0(6) $Å^3$	
Z	2	
Density (calculated)	1.293 Mg/m^3	
Absorption coefficient	0.086 mm^{-1}	
F(000)	468	
Crystal size	0.21 x 0.16 x 0.10 mm^3	
Theta range for data collection	1.82 to 27.46°.	
Index ranges	-10<=h<=11, -15<=k<=15, -15<=l<=15	
Reflections collected	17503	
Independent reflections	5008 [R(int) = 0.0746]	
Completeness to theta = 27.46°	95.3 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7455 and 0.5812	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5008 / 8 / 284	
Goodness-of-fit on F^2	1.528	
Final R indices [I>2sigma(I)]	R1 = 0.1450, wR2 = 0.4332	
R indices (all data)	R1 = 0.2083, wR2 = 0.4664	
Extinction coefficient	0.07(2)	
Largest diff. peak and hole	0.552 and -0.407 $e.Å^{-3}$	

Table 6-2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
For formamide **3** sk_ano2bm_cn_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2504(8)	-236(6)	10037(7)	73(2)
C(2)	1769(8)	698(6)	9069(7)	71(2)
C(3)	1831(7)	1900(5)	8826(6)	63(2)
C(4)	2638(6)	2181(5)	9599(5)	50(1)
C(5)	3423(7)	1203(5)	10594(5)	63(2)
C(6)	3358(8)	-15(6)	10812(6)	79(2)
C(7)	1509(5)	4437(4)	8841(4)	40(1)
C(8)	-52(5)	4339(5)	8903(4)	46(1)
C(9)	-1213(6)	5384(5)	8446(5)	52(1)
C(10)	-867(6)	6553(5)	7985(5)	56(1)
C(11)	667(6)	6676(5)	7908(4)	51(1)
C(12)	1851(5)	5640(4)	8333(4)	38(1)
C(13)	4468(6)	5587(5)	7360(5)	55(1)
C(14)	3561(6)	6071(4)	6157(4)	72(2)
C(15)	3202(6)	5211(5)	5767(4)	96(3)
C(16)	3584(7)	3918(5)	6291(5)	119(4)
C(17)	3083(8)	3236(7)	5754(6)	181(7)
C(18)	2201(8)	3848(9)	4693(6)	223(11)
C(19)	1820(7)	5141(9)	4169(5)	222(11)
C(20)	2321(7)	5823(7)	4706(5)	176(7)
C(21)	1766(14)	7087(7)	4034(8)	230(12)
C(22)	2212(9)	7836(8)	4520(5)	182(8)
C(23)	1712(10)	9114(8)	3901(6)	238(12)
C(24)	2144(12)	9925(5)	4312(8)	253(11)
C(25)	3075(11)	9457(5)	5343(8)	206(7)
C(26)	3575(8)	8179(5)	5962(5)	133(4)
C(27)	3144(7)	7369(4)	5550(4)	98(3)

C(29)	3932(6)	6256(4)	8956(5)	49(1)
N(1)	2452(9)	-1542(7)	10316(9)	106(3)
N(2)	2710(4)	3392(3)	9367(4)	46(1)
N(3)	3441(4)	5785(3)	8269(3)	41(1)
O(1)	3141(10)	-2379(5)	11169(7)	145(3)
O(2)	1658(8)	-1695(5)	9581(8)	137(3)
O(3)	5236(4)	6509(4)	8926(4)	64(1)

Table 6-3. Bond lengths [Å] and angles [°] for formamide **3** sk_ano2bm_cn_0m.

C(1)-C(2)	1.335(10)
C(1)-C(6)	1.383(10)
C(1)-N(1)	1.473(9)
C(2)-C(3)	1.363(8)
C(2)-H(2)	0.9300
C(3)-C(4)	1.400(7)
C(3)-H(3)	0.9300
C(4)-N(2)	1.378(6)
C(4)-C(5)	1.390(8)
C(5)-C(6)	1.390(9)
C(5)-H(5)	0.9300
C(6)-H(6)	0.9300
C(7)-C(8)	1.384(6)
C(7)-N(2)	1.404(6)
C(7)-C(12)	1.407(6)
C(8)-C(9)	1.374(7)
C(8)-H(8)	0.9300
C(9)-C(10)	1.373(8)
C(9)-H(9)	0.9300
C(10)-C(11)	1.368(7)
C(10)-H(10)	0.9300
C(11)-C(12)	1.376(7)
C(11)-H(11)	0.9300
C(12)-N(3)	1.425(5)
C(13)-N(3)	1.456(6)
C(13)-C(14)	1.547(7)
C(13)-H(13A)	0.9700
C(13)-H(13B)	0.9700
C(14)-C(15)	1.401(8)
C(14)-C(27)	1.402(7)
C(15)-C(16)	1.3900
C(15)-C(20)	1.3900
C(16)-C(17)	1.3900
C(16)-H(16)	0.9300

C(17)-C(18)	1.3900
C(17)-H(17)	0.9300
C(18)-C(19)	1.3900
C(18)-H(18)	0.9300
C(19)-C(20)	1.3900
C(19)-H(19)	0.9300
C(20)-C(21)	1.392(12)
C(21)-C(22)	1.393(13)
C(21)-H(21)	0.9300
C(22)-C(23)	1.3900
C(22)-C(27)	1.3900
C(23)-C(24)	1.3900
C(23)-H(23)	0.9300
C(24)-C(25)	1.3900
C(24)-H(24)	0.9300
C(25)-C(26)	1.3900
C(25)-H(25)	0.9300
C(26)-C(27)	1.3900
C(26)-H(26)	0.9300
C(29)-O(3)	1.230(6)
C(29)-N(3)	1.326(6)
C(29)-H(29)	0.9300
N(1)-O(1)	1.197(10)
N(1)-O(2)	1.263(10)
N(2)-H(2A)	0.8600

C(2)-C(1)-C(6)	121.2(5)
C(2)-C(1)-N(1)	121.4(7)
C(6)-C(1)-N(1)	117.4(8)
C(1)-C(2)-C(3)	120.6(6)
C(1)-C(2)-H(2)	119.7
C(3)-C(2)-H(2)	119.7
C(2)-C(3)-C(4)	120.7(6)
C(2)-C(3)-H(3)	119.7
C(4)-C(3)-H(3)	119.7
N(2)-C(4)-C(5)	120.1(5)

N(2)-C(4)-C(3)	121.4(5)
C(5)-C(4)-C(3)	118.4(5)
C(4)-C(5)-C(6)	119.7(6)
C(4)-C(5)-H(5)	120.2
C(6)-C(5)-H(5)	120.2
C(1)-C(6)-C(5)	119.4(6)
C(1)-C(6)-H(6)	120.3
C(5)-C(6)-H(6)	120.3
C(8)-C(7)-N(2)	121.8(4)
C(8)-C(7)-C(12)	117.7(4)
N(2)-C(7)-C(12)	120.2(4)
C(9)-C(8)-C(7)	120.7(4)
C(9)-C(8)-H(8)	119.6
C(7)-C(8)-H(8)	119.6
C(10)-C(9)-C(8)	121.0(5)
C(10)-C(9)-H(9)	119.5
C(8)-C(9)-H(9)	119.5
C(11)-C(10)-C(9)	119.3(5)
C(11)-C(10)-H(10)	120.3
C(9)-C(10)-H(10)	120.3
C(10)-C(11)-C(12)	120.6(5)
C(10)-C(11)-H(11)	119.7
C(12)-C(11)-H(11)	119.7
C(11)-C(12)-C(7)	120.7(4)
C(11)-C(12)-N(3)	119.8(4)
C(7)-C(12)-N(3)	119.5(4)
N(3)-C(13)-C(14)	110.8(4)
N(3)-C(13)-H(13A)	109.5
C(14)-C(13)-H(13A)	109.5
N(3)-C(13)-H(13B)	109.5
C(14)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	108.1
C(15)-C(14)-C(27)	125.2(5)
C(15)-C(14)-C(13)	119.1(4)
C(27)-C(14)-C(13)	115.7(4)
C(16)-C(15)-C(20)	120.0

C(16)-C(15)-C(14)	129.5(3)
C(20)-C(15)-C(14)	110.5(3)
C(17)-C(16)-C(15)	120.0
C(17)-C(16)-H(16)	120.0
C(15)-C(16)-H(16)	120.0
C(18)-C(17)-C(16)	120.0
C(18)-C(17)-H(17)	120.0
C(16)-C(17)-H(17)	120.0
C(17)-C(18)-C(19)	120.0
C(17)-C(18)-H(18)	120.0
C(19)-C(18)-H(18)	120.0
C(18)-C(19)-C(20)	120.0
C(18)-C(19)-H(19)	120.0
C(20)-C(19)-H(19)	120.0
C(19)-C(20)-C(15)	120.0
C(19)-C(20)-C(21)	109.3(5)
C(15)-C(20)-C(21)	130.7(5)
C(20)-C(21)-C(22)	112.8(6)
C(20)-C(21)-H(21)	123.6
C(22)-C(21)-H(21)	123.6
C(23)-C(22)-C(27)	120.0
C(23)-C(22)-C(21)	116.6(6)
C(27)-C(22)-C(21)	123.4(6)
C(22)-C(23)-C(24)	120.0
C(22)-C(23)-H(23)	120.0
C(24)-C(23)-H(23)	120.0
C(25)-C(24)-C(23)	120.0
C(25)-C(24)-H(24)	120.0
C(23)-C(24)-H(24)	120.0
C(24)-C(25)-C(26)	120.0
C(24)-C(25)-H(25)	120.0
C(26)-C(25)-H(25)	120.0
C(27)-C(26)-C(25)	120.0
C(27)-C(26)-H(26)	120.0
C(25)-C(26)-H(26)	120.0
C(26)-C(27)-C(22)	120.0

C(26)-C(27)-C(14)	122.7(4)
C(22)-C(27)-C(14)	117.2(5)
O(3)-C(29)-N(3)	125.4(5)
O(3)-C(29)-H(29)	117.3
N(3)-C(29)-H(29)	117.3
O(1)-N(1)-O(2)	123.8(7)
O(1)-N(1)-C(1)	121.2(9)
O(2)-N(1)-C(1)	115.0(9)
C(4)-N(2)-C(7)	125.8(4)
C(4)-N(2)-H(2A)	117.1
C(7)-N(2)-H(2A)	117.1
C(29)-N(3)-C(12)	119.0(4)
C(29)-N(3)-C(13)	119.2(4)
C(12)-N(3)-C(13)	121.3(4)

Symmetry transformations used to generate equivalent atoms:

Crystal data for formamide derivative 5 (with pyrene)

Table 7-1. Crystal data and structure refinement for formamide **5** py_cn_0m. **CCDC no. 916000**

Identification code	shelxl	
Empirical formula	C32 H27 N3 O4 S	
Formula weight	549.63	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P21/c	
Unit cell dimensions	a = 12.604(8) Å	□= 90°.
	b = 9.138(4) Å	□= 97.91(2)°.
	c = 23.915(16) Å	□ = 90°.
Volume	2728(3) $Å^3$	
Z	4	
Density (calculated)	1.338 Mg/m^3	
Absorption coefficient	0.162 mm^{-1}	
F(000)	1152	
Crystal size	0.21 x 0.18 x 0.12 mm^3	
Theta range for data collection	1.63 to 27.26°.	
Index ranges	-15<=h<=16, -11<=k<=11, -30<=l<=30	
Reflections collected	17751	
Independent reflections	5828 [R(int) = 0.0528]	
Completeness to theta = 27.26°	95.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7455 and 0.600	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5828 / 2 / 369	
Goodness-of-fit on F^2	1.034	
Final R indices [I>2sigma(I)]	R1 = 0.0687, wR2 = 0.1574	
R indices (all data)	R1 = 0.1377, wR2 = 0.1955	
Largest diff. peak and hole	0.419 and -0.388 $e.Å^{-3}$	

Table 7-2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)

For formamide **5** py_cn_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3912(3)	1742(5)	9808(1)	73(1)
C(2)	3157(3)	773(4)	9945(1)	74(1)
C(3)	2843(3)	814(4)	10477(1)	60(1)
C(4)	3268(2)	1867(3)	10866(1)	43(1)
C(5)	4004(2)	2863(4)	10708(1)	53(1)
C(6)	4321(3)	2810(4)	10181(1)	68(1)
C(7)	2348(2)	1000(3)	11652(1)	39(1)
C(8)	2557(2)	-492(3)	11651(1)	50(1)
C(9)	1957(3)	-1467(3)	11912(1)	56(1)
C(10)	1146(3)	-974(4)	12187(1)	60(1)
C(11)	932(2)	499(3)	12203(1)	52(1)
C(12)	1532(2)	1500(3)	11943(1)	41(1)
C(13)	331(2)	3544(4)	11572(1)	57(1)
C(14)	391(2)	3219(3)	10963(1)	51(1)
C(15)	-259(3)	2116(4)	10703(1)	64(1)
C(16)	-263(3)	1784(4)	10142(2)	68(1)
C(17)	370(3)	2524(4)	9816(1)	62(1)
C(18)	395(3)	2212(5)	9232(2)	80(1)
C(19)	1010(3)	2954(5)	8926(2)	84(1)
C(20)	1677(3)	4136(5)	9152(1)	74(1)
C(21)	2301(4)	4984(7)	8847(2)	98(2)
C(22)	2913(4)	6122(6)	9083(2)	100(2)
C(23)	2933(3)	6467(5)	9652(2)	83(1)
C(24)	2329(3)	5651(4)	9985(1)	63(1)
C(25)	2330(3)	5956(4)	10577(1)	61(1)
C(26)	1723(2)	5184(3)	10884(1)	52(1)
C(27)	1046(2)	4018(3)	10647(1)	46(1)

C(28)	1047(2)	3678(3)	10066(1)	51(1)
C(29)	1688(3)	4490(4)	9740(1)	57(1)
C(30)	1762(2)	3905(3)	12362(1)	47(1)
C(31)	4207(3)	6305(4)	12845(1)	80(1)
C(32)	5591(3)	6185(4)	12100(2)	74(1)
N(1)	4263(4)	1648(6)	9254(2)	114(1)
N(2)	2964(2)	2002(3)	11392(1)	44(1)
N(3)	1275(2)	3030(2)	11955(1)	42(1)
O(1)	5006(3)	2451(5)	9163(1)	144(2)
O(2)	3626(14)	1004(18)	8867(5)	132(3)
O(2')	4110(20)	570(30)	8976(8)	132(3)
O(3)	1546(2)	5192(3)	12426(1)	66(1)
O(4)	4057(2)	4275(2)	12066(1)	69(1)
S(1)	4225(1)	5884(1)	12122(1)	54(1)

Table 7-3. Bond lengths [Å] and angles [°] for formamide **5** py_cn_0m.

C(1)-C(2)	1.372(5)
C(1)-C(6)	1.374(5)
C(1)-N(1)	1.456(5)
C(2)-C(3)	1.383(5)
C(2)-H(2)	0.9300
C(3)-C(4)	1.394(4)
C(3)-H(3)	0.9300
C(4)-N(2)	1.371(3)
C(4)-C(5)	1.389(4)
C(5)-C(6)	1.374(4)
C(5)-H(5)	0.9300
C(6)-H(6)	0.9300
C(7)-C(8)	1.389(4)
C(7)-C(12)	1.394(4)
C(7)-N(2)	1.400(3)
C(8)-C(9)	1.372(4)
C(8)-H(8)	0.9300
C(9)-C(10)	1.365(4)
C(9)-H(9)	0.9300
C(10)-C(11)	1.374(4)
C(10)-H(10)	0.9300
C(11)-C(12)	1.388(4)
C(11)-H(11)	0.9300
C(12)-N(3)	1.436(3)
C(13)-N(3)	1.474(4)
C(13)-C(14)	1.500(4)
C(13)-H(13A)	0.9700
C(13)-H(13B)	0.9700
C(14)-C(15)	1.390(5)
C(14)-C(27)	1.397(4)
C(15)-C(16)	1.376(5)
C(15)-H(15)	0.9300
C(16)-C(17)	1.368(5)
C(16)-H(16)	0.9300

C(17)-C(18)	1.432(5)
C(17)-C(28)	1.434(5)
C(18)-C(19)	1.322(6)
C(18)-H(18)	0.9300
C(19)-C(20)	1.428(6)
C(19)-H(19)	0.9300
C(20)-C(21)	1.382(6)
C(20)-C(29)	1.441(4)
C(21)-C(22)	1.369(7)
C(21)-H(21)	0.9300
C(22)-C(23)	1.394(6)
C(22)-H(22)	0.9300
C(23)-C(24)	1.391(5)
C(23)-H(23)	0.9300
C(24)-C(29)	1.410(5)
C(24)-C(25)	1.443(5)
C(25)-C(26)	1.332(4)
C(25)-H(25)	0.9300
C(26)-C(27)	1.432(4)
C(26)-H(26)	0.9300
C(27)-C(28)	1.424(4)
C(28)-C(29)	1.409(5)
C(30)-O(3)	1.222(4)
C(30)-N(3)	1.341(4)
C(30)-H(30)	0.9300
C(31)-S(1)	1.773(3)
C(31)-H(31A)	0.9600
C(31)-H(31B)	0.9600
C(31)-H(31C)	0.9600
C(32)-S(1)	1.753(4)
C(32)-H(32A)	0.9600
C(32)-H(32B)	0.9600
C(32)-H(32C)	0.9600
N(1)-O(2')	1.19(2)
N(1)-O(1)	1.232(5)
N(1)-O(2)	1.281(16)

N(2)-H(2A)	0.860(18)
O(4)-S(1)	1.488(2)

C(2)-C(1)-C(6)	120.9(3)
C(2)-C(1)-N(1)	119.2(4)
C(6)-C(1)-N(1)	119.9(4)
C(1)-C(2)-C(3)	119.9(3)
C(1)-C(2)-H(2)	120.1
C(3)-C(2)-H(2)	120.1
C(2)-C(3)-C(4)	120.0(3)
C(2)-C(3)-H(3)	120.0
C(4)-C(3)-H(3)	120.0
N(2)-C(4)-C(5)	118.2(3)
N(2)-C(4)-C(3)	123.0(3)
C(5)-C(4)-C(3)	118.7(3)
C(6)-C(5)-C(4)	121.0(3)
C(6)-C(5)-H(5)	119.5
C(4)-C(5)-H(5)	119.5
C(5)-C(6)-C(1)	119.4(3)
C(5)-C(6)-H(6)	120.3
C(1)-C(6)-H(6)	120.3
C(8)-C(7)-C(12)	118.4(3)
C(8)-C(7)-N(2)	121.6(2)
C(12)-C(7)-N(2)	119.9(3)
C(9)-C(8)-C(7)	121.4(3)
C(9)-C(8)-H(8)	119.3
C(7)-C(8)-H(8)	119.3
C(10)-C(9)-C(8)	120.0(3)
C(10)-C(9)-H(9)	120.0
C(8)-C(9)-H(9)	120.0
C(9)-C(10)-C(11)	120.0(3)
C(9)-C(10)-H(10)	120.0
C(11)-C(10)-H(10)	120.0
C(10)-C(11)-C(12)	120.7(3)
C(10)-C(11)-H(11)	119.6
C(12)-C(11)-H(11)	119.6

C(11)-C(12)-C(7)	119.5(3)
C(11)-C(12)-N(3)	119.6(2)
C(7)-C(12)-N(3)	120.9(2)
N(3)-C(13)-C(14)	113.3(2)
N(3)-C(13)-H(13A)	108.9
C(14)-C(13)-H(13A)	108.9
N(3)-C(13)-H(13B)	108.9
C(14)-C(13)-H(13B)	108.9
H(13A)-C(13)-H(13B)	107.7
C(15)-C(14)-C(27)	119.6(3)
C(15)-C(14)-C(13)	118.1(3)
C(27)-C(14)-C(13)	122.2(3)
C(16)-C(15)-C(14)	121.2(3)
C(16)-C(15)-H(15)	119.4
C(14)-C(15)-H(15)	119.4
C(17)-C(16)-C(15)	121.5(3)
C(17)-C(16)-H(16)	119.3
C(15)-C(16)-H(16)	119.3
C(16)-C(17)-C(18)	123.4(4)
C(16)-C(17)-C(28)	119.1(3)
C(18)-C(17)-C(28)	117.5(4)
C(19)-C(18)-C(17)	122.1(4)
C(19)-C(18)-H(18)	119.0
C(17)-C(18)-H(18)	119.0
C(18)-C(19)-C(20)	122.5(3)
C(18)-C(19)-H(19)	118.7
C(20)-C(19)-H(19)	118.7
C(21)-C(20)-C(19)	124.9(4)
C(21)-C(20)-C(29)	117.3(4)
C(19)-C(20)-C(29)	117.7(4)
C(22)-C(21)-C(20)	122.6(4)
C(22)-C(21)-H(21)	118.7
C(20)-C(21)-H(21)	118.7
C(21)-C(22)-C(23)	120.4(4)
C(21)-C(22)-H(22)	119.8
C(23)-C(22)-H(22)	119.8

C(24)-C(23)-C(22)	120.0(4)
C(24)-C(23)-H(23)	120.0
C(22)-C(23)-H(23)	120.0
C(23)-C(24)-C(29)	119.6(3)
C(23)-C(24)-C(25)	122.0(4)
C(29)-C(24)-C(25)	118.4(3)
C(26)-C(25)-C(24)	121.0(3)
C(26)-C(25)-H(25)	119.5
C(24)-C(25)-H(25)	119.5
C(25)-C(26)-C(27)	122.2(3)
C(25)-C(26)-H(26)	118.9
C(27)-C(26)-H(26)	118.9
C(14)-C(27)-C(28)	119.5(3)
C(14)-C(27)-C(26)	122.7(3)
C(28)-C(27)-C(26)	117.8(3)
C(29)-C(28)-C(27)	120.2(3)
C(29)-C(28)-C(17)	120.6(3)
C(27)-C(28)-C(17)	119.1(3)
C(28)-C(29)-C(24)	120.4(3)
C(28)-C(29)-C(20)	119.5(3)
C(24)-C(29)-C(20)	120.1(3)
O(3)-C(30)-N(3)	125.2(3)
O(3)-C(30)-H(30)	117.4
N(3)-C(30)-H(30)	117.4
S(1)-C(31)-H(31A)	109.5
S(1)-C(31)-H(31B)	109.5
H(31A)-C(31)-H(31B)	109.5
S(1)-C(31)-H(31C)	109.5
H(31A)-C(31)-H(31C)	109.5
H(31B)-C(31)-H(31C)	109.5
S(1)-C(32)-H(32A)	109.5
S(1)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5
S(1)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5

O(2')-N(1)-O(1)	117.7(13)
O(1)-N(1)-O(2)	124.2(7)
O(2')-N(1)-C(1)	120.7(13)
O(1)-N(1)-C(1)	117.3(5)
O(2)-N(1)-C(1)	116.5(8)
C(4)-N(2)-C(7)	126.2(2)
C(4)-N(2)-H(2A)	114(2)
C(7)-N(2)-H(2A)	120(2)
C(30)-N(3)-C(12)	120.9(2)
C(30)-N(3)-C(13)	120.7(2)
C(12)-N(3)-C(13)	117.5(2)
O(4)-S(1)-C(32)	106.26(17)
O(4)-S(1)-C(31)	106.43(16)
C(32)-S(1)-C(31)	98.05(18)

Symmetry transformations used to generate equivalent atoms: