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SYNTHESIS, AND CRYSTAL STRUCTURE OF TRIMETHYLAMMONIUM 5-(5-CHLORO-2,4-DINITROPHENYL)- 2,6-DIOXO-1,2,3,6-TETRAHYDROPYRIMIDIN-4-OLATE

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ABSTRACT

A family of derivatives of barbituric acid, barbiturates have a long history of medicinal use as a variety of sedatives, hypnotics, anti-epileptics, sleep aids, and muscle relaxants. They have also been used in anaesthesia and to map brain dysfunction prior to brain surgery. A novel stable carbanionic sigma complex(CSC) with excellent purity and yield was synthesised in this work by combining DCDNB: 1,3-dichloro-4,6-dinitrobenzene, barbituric acid: pyrimidine-2,4,6(1H,3H,5H)-trione, and ethanol as base. The new carbanionic sigma complex is the molecular salt (barbiturates). The compound has been characterised by IR, 1H NMR and 13C NMR. The proposed structure has also been confirmed by single-crystal XRD studies.

Keywords: DCDNB, Barbituric Acid, Trimethylamine, FT-IR,^{1}H-NMR, XRD,^{13}C-NMR

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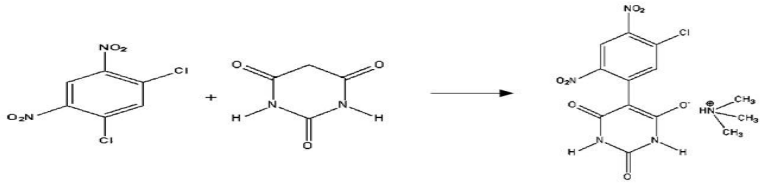
INTRODUCTION

A wide variety of products, including pharmaceuticals, colours, high-energy explosives, and dyes[1-5], are produced using nitroaromatic compounds, which are a result of nitro group chemistry. When these electron-deficient nitroaromatic compounds react rapidly with electron-rich molecules, then strong colours are produced.[6,7] There are three main types of interactions that can occur between electron-rich molecules and electron-deficient nitroaromatic compounds that can lead to the formation of complexes such as radical anion and anionic sigma (σ) complexes.[8,9] Nucleophiles may create anionic σ complexes as permanent or transitory entities by covalently bonding to a substituted ring carbon of aromatic and heteroaromatic electron-deficient substrates. It has been known since 1900, when Jackson and Gazzolo introduced their technique, that picryl ethers react with potassium alkoxides to produce reactive chemicals.[10] In 1902, Meisenheimer found the first chemical evidence of this structure, which led to the widespread naming of these compounds as "Jackson-Meisenheimer" or "Meisenheimer" complexes.[11] 1,3-Dichloro-4,6-dinitrobenzene (DCDNB) has been a precursor for many products in recent times. We have learned about many dinitro and diamino benzene compounds with added substituents from DCDNB.[12-16] The present work focuses on the development and pharmacological evaluation of barbituric acid derivatives with potential anticonvulsant and central nervous system activity, contributing to therapeutic advancements in neurological disorders while promoting inclusive and equitable healthcare opportunities in line with Sustainable Development Goal-3 (Good health and well-being).

EXPERIMENTAL

The temperature was raised to 303 K in a 30-mL and 50-mL 100% ethanol solution that included 2.37 g of DCDNB and 0.01 mol of BA. The mixture was rapidly stirred for two to three hours after 3 millilitres of trimethylamine (~0.05 mol) was added to the solution. Crystals of a maroon-red hue will form in the ethanolic solution after one week of storage, after which the surplus ethanol will be distilled off using decreased pressure. The crystals were purified by filtering, grinding, and washing them with a large quantity of ether to eliminate any remaining impurities. They were then recrystallised from a combination of DMSO and ethanol, with a yield of 80% and a melting point of 573 K.



Scheme-1

RESULTS AND DISCUSSION

Single Crystal XRD of CSC

The single crystal XRD confirms the presence of DCDNB, BA, and trimethylamine in the carbanionic σ complex[25]. In Tables 2–5, you may find the crystallographic data, specified bond lengths, chosen bond angles, and hydrogen bonds, in that order. In Fig.-1 we can see the ORTEP complex. Figure-2 displays the algebraic representation of the carbanionic σ complex. Two sets of cation and anion units, together with a solvent molecule (DMSO), make up the asymmetric unit. It crystallises in a triclinic P-1 system. The barbiturate molecules related by inversion are joined together by Ƀ2(8) rings in the following sequence: N-H…O (N5-H5A…O10 and N6-H6A…O1). A hydrogen bond is formed between the barbiturate unit and DMSO that has N3-H3A-O15. There is a hydrogen connection between the anion and the cation residues with N9-H9A…O3..Anions have a dihedral angle of 40.03 (7) Ϙ and 40.80 (6) between their rings. The ortho-substituted nitro group's plane, when measured against the barbiturate ring, is 42.83 Ğ away from the aromatic plane.

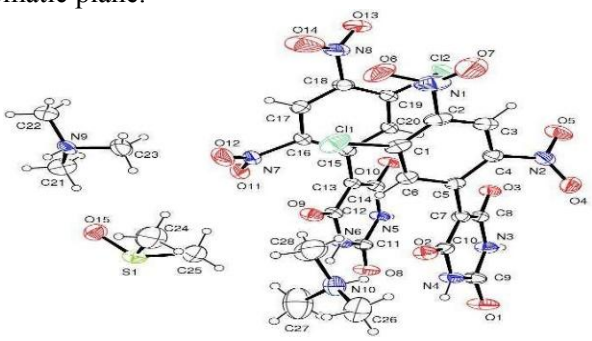


Fig.-1: View of the CSC from ORTEP

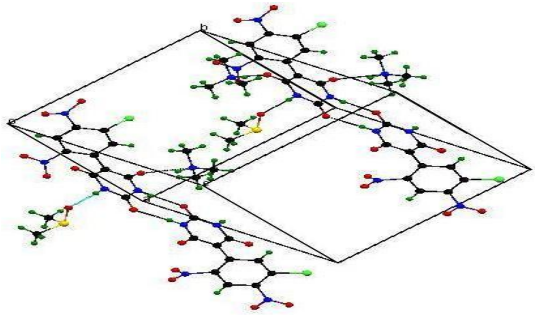


Fig.-2: Crystal Packing Pattern of CSC

Table-1: Physical And Visible Spectral Data of Carbanionic Σ Complex

Data	Complex
Molecular formula	$C_{13}H_{14}N_5O_7Cl$
Molecular weight	387.5
Physical state	Maroon red crystalline solid
Stability	Stable under ordinary condition. Decomposes if heated to high temperature
Melting point	> 573 K
Yield	80%
Solubility/100 ml (i) H_2O (ii) EtOH (iii) DMSO	 2.33 g 2.60 g 9.82 g
λ_{max} (H_2O)	400 nm
ε_{max} (H_2O)	4420 l $mol^{-1}cm^{-1}$
λ_{max} (EtOH)	430 nm
ε_{max} (EtOH)	12480 l $mol^{-1}cm^{-1}$
λ_{max} (DMSO)	485 nm
ε_{max} (DMSO)	13180 l $mol^{-1}cm^{-1}$

Table-2: Crystal Data and Structure Refinement Parameters for the Synthesized Compound

Parameter	Value
Empirical formula	$C_{28}H_{34}Cl_2N_{10}O_{15}$
Formula weight	853.61
Temperature (K)	293(2)
Crystal system	Triclinic
Space group	*P*–1
a (Å)	12.2380(3)
b (Å)	12.2560(3)
c (Å)	13.5470(3)
α (°)	74.0540(10)
β (°)	79.0790(10)
γ (°)	85.7740(10)
Volume (Å^3)	1917.83(8)
Z	2
Calculated density (Mg m^{-3}) Absorption coefficient, μ (mm^{-1})	1.478 0.304
Crystal size (mm^3)	0.30 × 0.20 × 0.20
θ range for data collection (°)	2.01–25.00
Index ranges	$-14 \le h \le 14$, $-14 \le k \le 14$, $-16 \le l \le 16$
Completeness to θ (%)	99.8
Reflections collected	33191
Independent reflections	6760
*R*int	0.0295
Data/restraints/parameters	627
Final *R* indices [I > 2σ(I)]	R_1 = 0.0404, wR_2 = 0.1015
R indices (all data)	R_1 = 0.0592, wR_2 = 0.1172
Largest diff. peak and hole (e Å^{-3})	0.359 and −0.353

Trimethylammonium 5-(5-chloro-2,4-dinitrophenyl)-2,6-dioxo-1,2,3,6-tetrahydropyrimidin-4-olate

A Spectroscopic Analysis

Figure-3 displays the infrared spectra of DCDNB. A strong and prominent absorption band at 832 cm^{-1} corresponds to the C–Cl stretching vibration of DCDNB.[17] The presence of this band at 833 cm^{-1} in the compound further substantiates the existence of the C–Cl bond[18] . Although DCDNB has two chlorine

atoms in a similar environment, only one is substituted during the formation of the CSC, as seen in Scheme-1. Figure-3 illustrates that the N–O bond in nitro groups accounts for the asymmetric stretching vibrational mode in DCDNB, whereas the symmetric stretching vibrational mode corresponds to the pronounced, sharp bands seen at 1345 cm^{-1}. The detection of these bands in the carbanionic σ complex IR spectra, seen in Fig.-6, indicates the existence of a nitroaromatic group. The CSC is presumably an amine salt, as indicated by the broad band seen in its infrared spectra (Fig.-6), which ranges from around 2400 to 3500 cm^{-1}. At around 524 cm^{-1}, a prominent and clear band is seen, indicating the torsional oscillation of the cationic component of the amine salt.[19] The C=C stretching frequency band appears at a lower frequency (~1633 cm^{-1}) than expected (1650 cm^{-1}) due to the delocalisation of negative charge in the complexes. Figure-5 indicates that the stretching frequency of the C–H bond in $N(CH_3)_3$ is around 2900 cm^{-1}. The protonation of the nitrogen atom during the formation of the carbanionic σ complex is shown by the signal shift to the high-frequency region of about 2990 cm^{-1} (Fig.-6).[20,21] Protonation reduces the length of the C–H bond, hence enhancing its strength. The infrared spectra of DCDNB, BA, trimethylamine, and CSC are shown in Fig.-3, 4, 5, and 6. The PMR spectra of DCDNB, BA, trimethylamine, and CSC are shown in Figures 7, 8, 9, and 10, respectively. Figures-11, 12, 13, and 14 illustrate the ^{13}C NMR spectra of DCDNB, BA, trimethylamine, and CSC, respectively. Figures-15 and 15a illustrate the HRMS of the CSC.[20,21] The structure shown in Scheme-1 is supported by spectral data. The CSC, in contrast, appears maroon-red and absorbs light at 430 nm, suggesting that the anion moiety's negative charge is dispersed across a large region. In contrast, DCDNB is light yellow and absorbs in the near UV-region (λmax = 255 nm). Results from qualitative analyses suggest the existence of nitrogen, nitrogen groups, and chlorine[21].

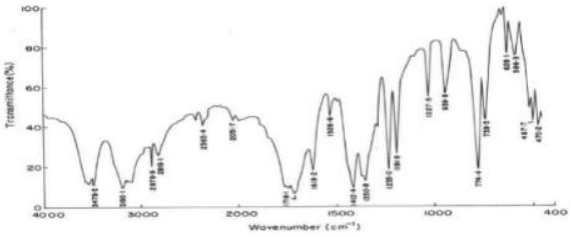


Fig.-3: DCDNB Infrared Spectrum

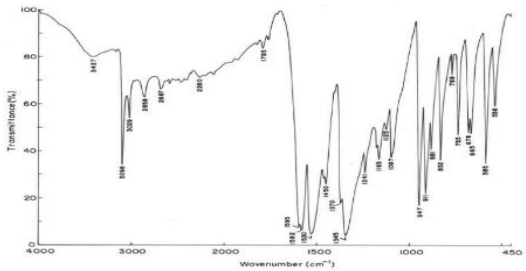


Fig.-4: Barbituric Acid (BA)- IR Spectra

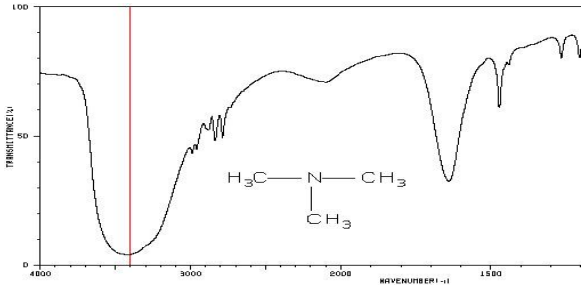


Fig.-5: Infrared Spectrum of Trimethylamine

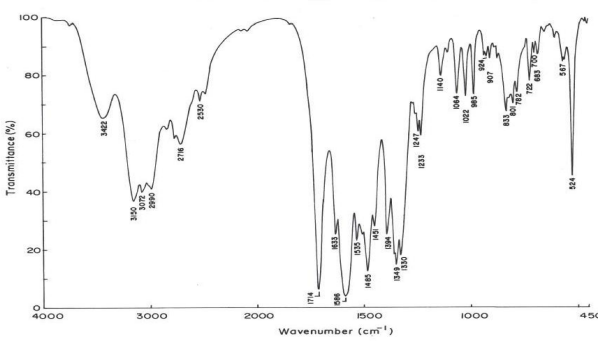


Fig.-6: CSC- IR Spectra

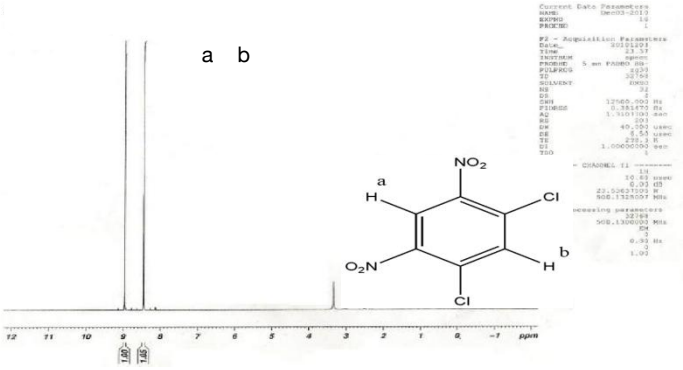


Fig.- 7: PMR Spectrum of DCDNB

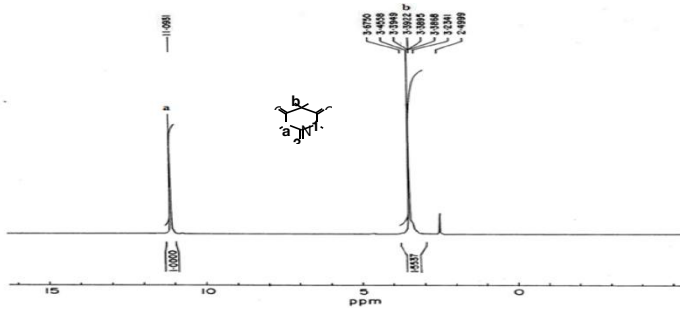


Fig.-8: PMR Spectrum of Barbituric Acid (BA)

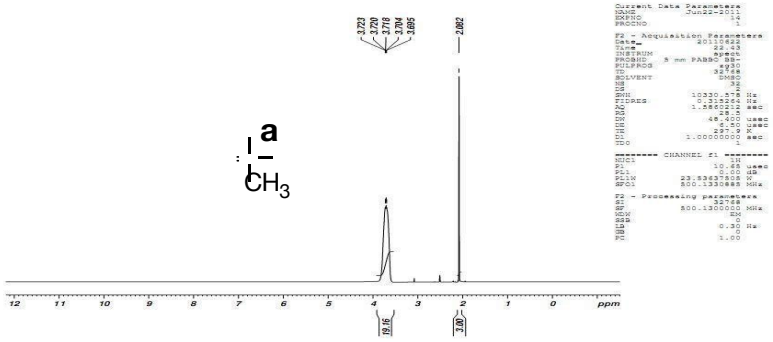


Fig.-9: PMR Spectrum of Trimethylamine

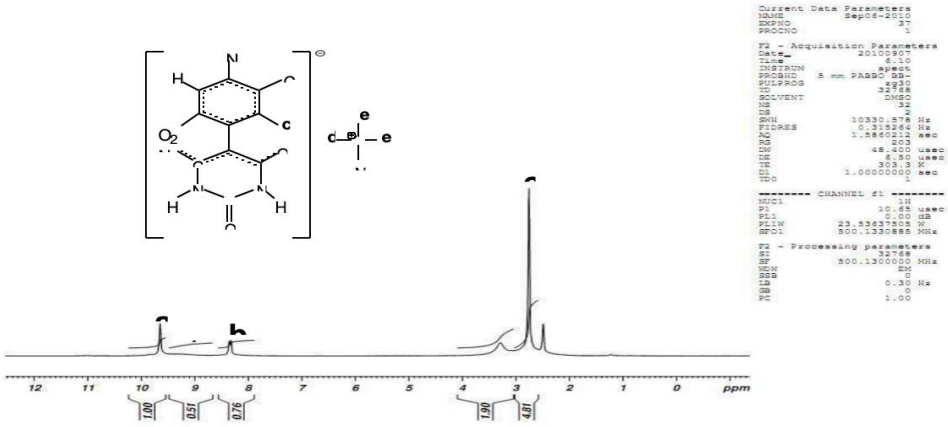


Fig.-10: PMR Spectrum of Carbanionic σ Complex

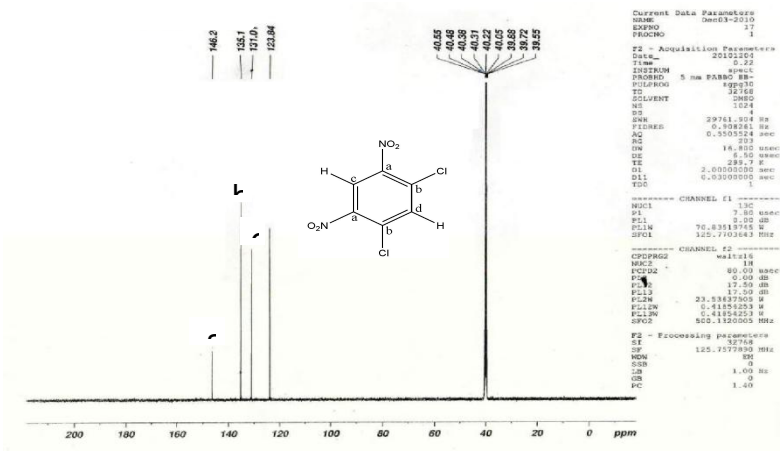


Fig.-11: ^{13}C NMR Spectrum of DCDNB

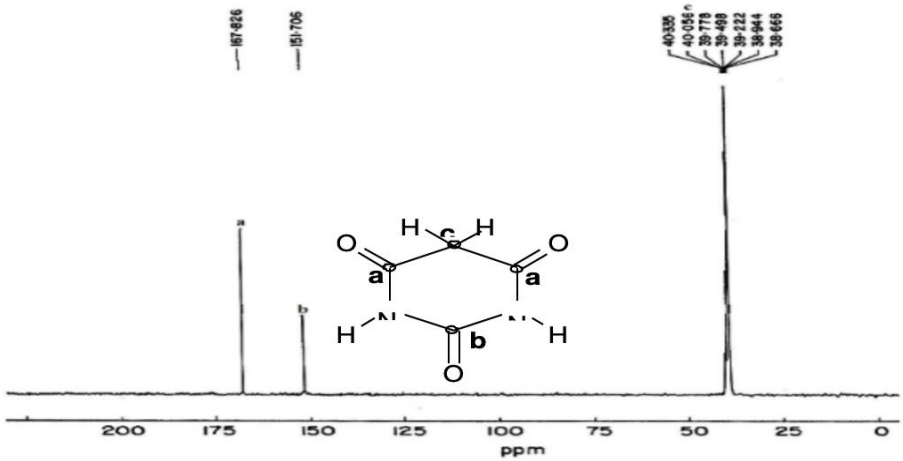


Fig.-12: ^{13}C NMR Spectrum of Barbituric Acid (BA)

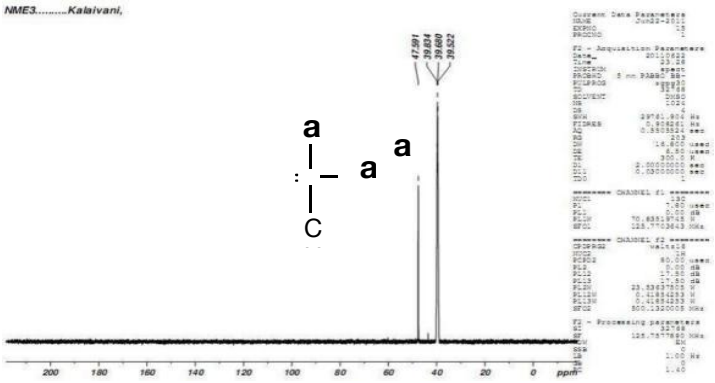


Fig.-13: ^{13}C NMR Spectrum of Trimethylamine

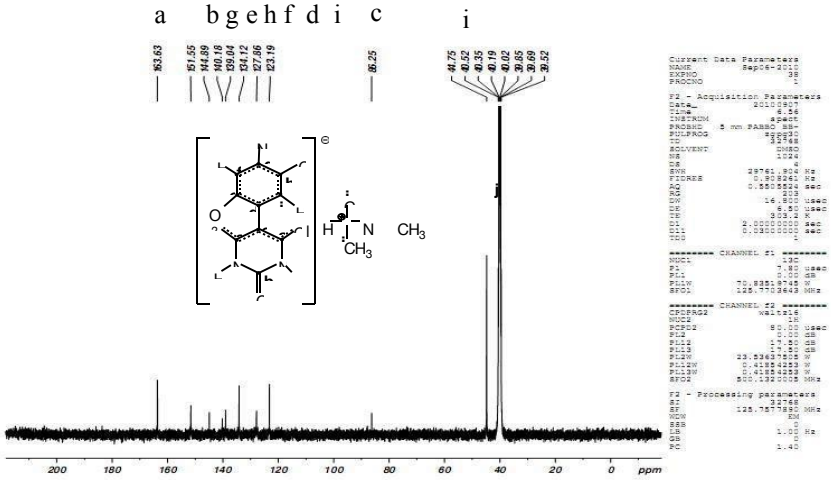


Fig.-14: ^{13}C NMR Spectrum of Carbanionic σ Complex

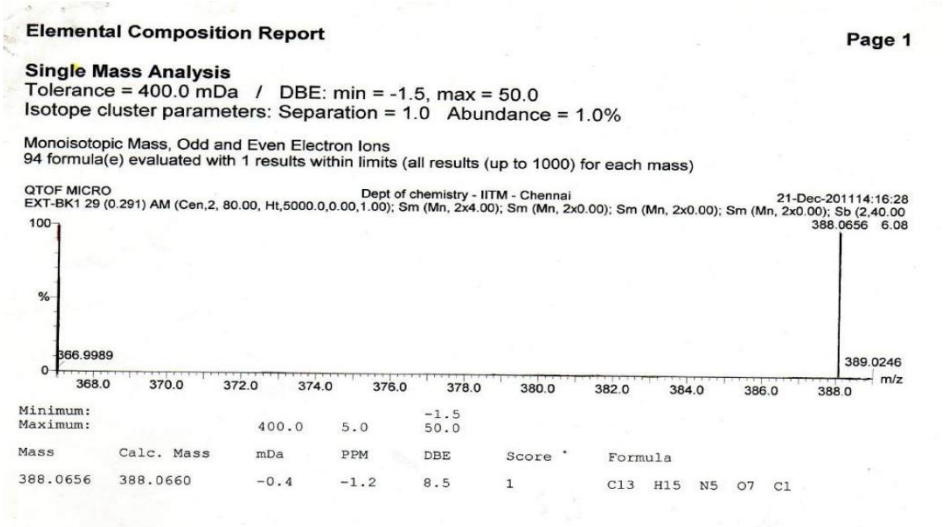


Fig.-15 High Resolution Mass Spectrum of CSC

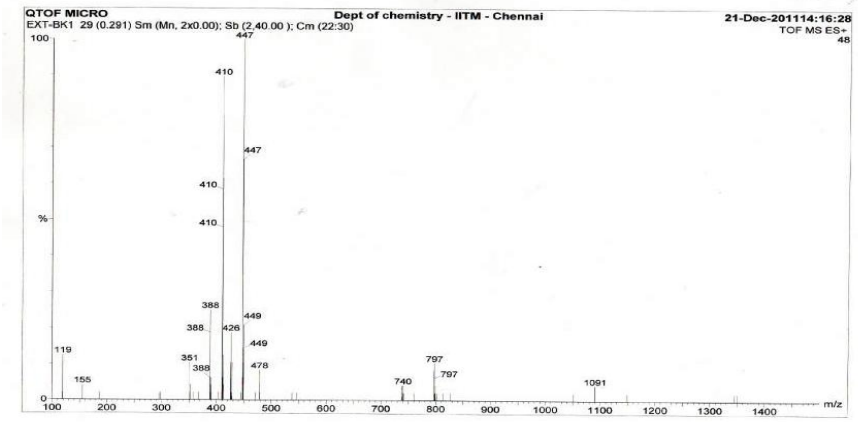


Fig.-15a: High Resolution Mass Spectrum of CSC

Table-2: Crystal data and structure refinement parameters for the synthesized compound.

Parameter	Value
Empirical formula	$C_{28}H_{34}Cl_2N_{10}O_{15}$
Formula weight	853.61
Temperature (K)	293(2)
Crystal system	Triclinic
Space group	*P*–1
a (Å)	12.2380(3)
b (Å)	12.2560(3)
c (Å)	13.5470(3)
α (°)	74.0540(10)
β (°)	79.0790(10)
γ (°)	85.7740(10)
Volume (Å³)	1917.83(8)
Z	2
Calculated density (Mg m^{-3})	1.478
Absorption coefficient, μ (mm^{-1})	0.304
Crystal size (mm^3)	0.30 × 0.20 × 0.20
θ range for data collection (°)	2.01–25.00
Index ranges	$-14 \le h \le 14$, $-14 \le k \le 14$, $-16 \le l \le 16$
Completeness to θ (%)	99.8
Reflections collected	33191
Independent reflections	6760
*R*int	0.0295
Data/restraints/parameters	627
Final *R* indices [I > 2σ(I)]	$R_1 = 0.0404$, w$R_2 = 0.1015$
R indices (all data)	$R_1 = 0.0592$, w$R_2 = 0.1172$
Largest diff. peak and hole (e $Å^{-3}$)	0.359 and −0.353

Table-3: Selected Bond Distance of The (A^0) of the CSC

Bond	Distance (Å)	Bond	Distance (Å)
C1 – C6	1.380(3)	C16 – N7	1.472(3)
C1 – C2	1.381(4)	C17 – C18	1.375(3)
C1 – Cl1	1.721(3)	C18 – C19	1.396(3)
C2 – C3	1.370(4)	C18 – N8	1.453(3)
C2 – N1	1.465(3)	C19 – C20	1.368(3)
C3 – C4	1.379(3)	C19 – Cl2	1.727(2)
C4 – C5	1.403(3)	C21 – N9	1.471(4)

C4 – N2	1.465(3)	C22 – N9	1.476(4)
C5 – C6	1.398(3)	C23 – N9	1.473(3)
C5 – C7	1.469(3)	C24 – S1	1.820(13)
C7 – C8	1.407(3)	C25 – S1	1.692(15)
C7 – C10	1.413(3)	C241 – S1	1.703(14)
C8 – O3	1.250(2)	C251 – S1	1.842(14)
C8 – N3	1.386(3)	C26 – N10	1.421(7)
C9 – O1	1.225(3)	C27 – N10	1.431(10)
C9 – N4	1.352(3)	C28 – N10	1.485(9)
C9 – N3	1.363(3)	C261 – N10	1.511(10)
C10 – O2	1.250(2)	C271 – N10	1.449(8)
C10 – N4	1.381(3)	C281 – N10	1.472(9)
C11 – O8	1.228(3)	N1 – O7	1.112(7)
C11 – N6	1.353(3)	N1 – O6′	1.133(6)
C11 – N5	1.361(3)	N1 – O7′	1.233(6)
C12 – O9	1.232(2)	N1 – O6	1.315(6)
C12 – N6	1.401(3)	N2 – O4	1.211(3)
C12 – C13	1.426(3)	N2 – O5	1.228(3)
C13 – C14	1.408(3)	N7 – O12	1.214(3)
C13 – C15	1.445(3)	N7 – O11	1.217(3)
C14 – O10	1.252(2)	N8 – O14	1.179(10)
C14 – N5	1.390(3)	N8 – O141	1.200(3)
C15 – C16	1.399(3)	N8 – O13	1.250(4)
C15 – C20	1.408(3)	N8 – O131	1.317(12)
C16 – C17	1.369(3)	S1 – O15	1.479(2)

Table-4: Selected Bond Angle (°) of the CSC

Bond angle	(°)	Bond angle	(°)
C6 – C1 – C2	119.1 (2)	O8 – C11 – N6	123.02 (19)
C6 – C1 – Cl1	117.1 (2)	O8 – C11 – N5	121.70 (2)
C2 – C1 – Cl1	123.8 (2)	N6 – C11 – N5	115.30 (19)
C3 – C2 – C1	120.3 (2)	O9 – C12 – N6	117.67 (18)
C3 – C2 – N1	116.3 (3)	O9 – C12 – C13	126.41 (19)
C1 – C2 – N1	123.3 (3)	N6 – C12 – C13	115.92 (18)
C2 – C3 – C4	119.7 (2)	C14 – C13 – C12	120.26 (18)
C3 – C4 – C5	122.5 (2)	C14 – C13 – C15	119.31 (17)
C3 – C4 – N2	114.9 (2)	C12 – C13 – C15	120.43 (18)
C5 – C4 – N2	122.5 (2)	O10 – C14 – N5	116.92 (18)
C6 – C5 – C4	115.3 (2)	O10 – C14 – C13	125.95 (19)
C6 – C5 – C7	119.17 (19)	N5 – C14 – C13	117.08 (18)
C4 – C5 – C7	125.5 (2)	C16 – C15 – C20	114.74 (19)
C1 – C6 – C5	123.0 (2)	C16 – C15 – C13	125.58 (19)
C8 – C7 – C10	119.64 (19)	C20 – C15 – C13	119.65 (19)
C8 – C7 – C5	120.89 (18)	C17 – C16 – C15	123.40 (2)
C10 – C7 – C5	119.44 (18)	C17 – C16 – N7	114.60 (2)
O3 – C8 – N3	117.75 (19)	C15 – C16 – N7	121.70 (2)
O3 – C8 – C7	124.98 (19)	C16 – C17 – C18	119.80 (2)
N3 – C8 – C7	117.26 (18)	C17 – C18 – C19	119.50 (2)
O1 – C9 – N4	123.0 (2)	C17 – C18 – N8	116.40 (2)
O1 – C9 – N3	122.3 (2)	C19 – C18 – N8	124.00 (2)
N4 – C9 – N3	114.7 (2)	C20 – C19 – C18	119.40 (2)
O2 – C10 – N4	117.6(18)	C20 – C19 – Cl2	117.26 (17)
O2 – C10 – C7	126.2 (2)	C18 – C19 – Cl2	123.30 (17)
N4 – C10 – C7	116.70 (18)	C19– C20 – C15	123.1 (2)

Table-5: Hydrogen Bond Geometry (Å, °) of Carbanionic Σ Complex

Hydrogen Bond Interaction	Bond Length D–H (Å)	Hydrogen Acceptor Distance H···A (Å)	Donor–Acceptor Distance D···A (Å)	Bond Angle D–H···A (°)
N(3)–H(3A)···O(15) (#1)	0.854(17)	2.069(18)	2.883(3)	159(2)
N(3)–H(3A)···S(1) (#1)	0.854(17)	3.020(2)	3.671(2)	135(2)
N(6)–H(6A)···O(1) (#2)	0.844(17)	2.058(17)	2.901(2)	177(2)
N(5)–H(5A)···O(10) (#3)	0.860(17)	1.982(19)	2.817(2)	163(3)
N(9)–H(9A)···O(3) (#4)	0.860(18)	1.827(19)	2.675(2)	169(3)
N(4)–H(4A)···O(8) (#2)	0.843(17)	1.984(18)	2.824(2)	174(2)
N(10)–H(10A)···O(2)	0.860(18)	1.850(2)	2.673(3)	159(2)

Symmetry transformations used to generate equivalent atoms:
#1 x, y–1, z; #2 –x + 1, –y, -z + 1; #3 –x, –y, –z+1; #4 x, y+1, z.

Acute Cytotoxicity, Hypnotic Activity, and Anticonvulsant Efficacy

The development of anticonvulsant medications has historically relied heavily on screening techniques and structural alterations.[22] Despite having pharmacological effect, barbituric acid does not significantly depress the central nervous system. Its anticonvulsant and hypnotic qualities, however, may be improved by adding alkyl or aryl substituents to the carbon atoms next to the carbonyl (C=O) groups.[1] It is well known that barbiturates work mainly by depressing the central nervous system, which results in sedative, muscle relaxant, and anxiolytic effects.[7] In this work, the hydrogen atoms of the methylene ($–CH_2$) group of barbituric acid were targeted in order to create a unique family of CSC derivatives. With a dosage of around 25mg/kg, the synthesised derivatives showed encouraging anticonvulsant action, successfully lowering the frequency and intensity of experimentally produced seizures (Table-6). Additionally, assessment of hypnotic activity showed that these substances were efficacious in inducing hypnosis in albino mice (Table-7).

According to acute toxicity assessments, the synthesised CSC derivatives fall under toxicity class 4[23], and the treated animals did not exhibit any notable behavioural changes. It was discovered that the LD_{50} values were more than 1500 mg/kg, indicating comparatively low acute toxicity. Furthermore, an evaluation of cytotoxicity against the human breast cancer cell ine MCF-7[24] revealed negligible cytotoxic effects, with IC_{20} values greater than 430 μM (Table-9).These results point to a positive safety profile and the possibility of further pharmacological research. However, as Table-10 illustrates, the assessed CSC derivatives showed no discernible antitubercular action. All things considered, the current research advances our knowledge of barbituric acid derivatives and their possible medical uses. However, further research is needed to clarify their modes of action and maximise their pharmacological effectiveness.

Table-6: Anticonvulsant Effect of CSC Derivatives on Seizure Phases in Experimental Animals

Treatment	Dose (mg/kg)	Flexion (s)	Extension (s)	Clonus (s)	Stupor (s)	Survival Outcome
Saline Control	5 mL/kg	12.0 ± 1.0	21.0 ± 1.5	35.0 ± 2.5	149.0 ± 1.0	Recovered
CSC Derivative	—	6.0 ± 1.2	2.0 ± 0.3	18.0 ± 1.4	177.0 ± 7.5	Recovered
Phenobarbitone	20	2.0 ± 0.2	0.0	15.0 ± 0.8	57.0 ± 3.9	Recovered

Values are expressed as mean ± SD (n = 6). Duration of seizure phases was measured in seconds. Statistical significance was observed at $P < 0.001$ compared with the control group.

Table-7: Hypnotic Activity of Synthesised CSC Derivatives in Experimental Animals

Treatment Group	Dose (mg/kg)	Time of Administration (min)	Loss of Righting Reflex (min)	Recovery Time (min)	Onset of Hypnosis * (min)	Duration of Hypnosis† (min)
Saline Control	0	—	—	—	—	—
CSC Derivative	100	0	36.8 ± 7.2	124.4 ± 8.7	36.8 ± 7.2	87.6 ± 5.4

* Onset of hypnosis = Time to loss of righting reflex after administration.
† Duration of hypnosis = Time interval between loss and recovery of righting reflex.

Values are expressed as mean ± SEM (n = 6). Statistical significance was observed at $P < 0.001$ compared with the control group.

Table-8: Antimalarial Efficacy of CSC

Sample	IC_{50}(µM)	CC_{50}(µM)	Selectivity Index (SI = CC_{50} / IC_{50})
CSC	>1.0	196.19	<196.19
Standard Drug	0.004	>200	>50000

Table-9: Cytotoxicity Result of CSC

Complex	IC_{50} (µmol/mol)
CSC	453.45 ± 12.22

Table-10: Antituberculosis Activity of CSC

Complex	Activity		Cytotoxicity
	MABA	BACTEC	
	>50 µM	IA	Nontoxic

CONCLUSION

The characterisation of carbanionic sigma complexes was the primary emphasis of this investigation. When reacting with a combination of BA (which contains an acidic hydrogen atom) and an aliphatic amine, DCDNB is anticipated to undergo aromatic nucleophilic substitution due to its polarised C-Cl bond. A novel CSC is formed in this study by following the aromatic nucleophilic substitution reaction with an enolisation process. The structure was dissected using FT-IR, ^{1}H NMR, and ^{13}C NMR spectroscopy. The current results of the single-crystal XRD analysis of carbanionic σ complexes are reviewed. Spectral data reveal the structure, and the single crystal XRD measurements confirm it. The crystallisation system of the CSC may be either triclinic or monoclinic. The inversion-related barbiturates are connected in the crystal via Ƀ2(8) patterns. The crystals also display a considerable number of weak hydrogen bonds between carbon and oxygen, as well as nitrogen and hydrogen.

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CONFLICT OF INTEREST

With respect to this article's publishing, the writers affirm that they are not involved in any conflict of interest.

AUTHOR CONTRIBUTIONS

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