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NEWER APPROACHES OF MONITORING ORGANIC REACTIONS BY USING 1, 1, DIPHENYL-2-PICRYLHYDRAZYL AS A STABLE OXIDANT

VIRAJ PARESHBHAI JATAKIYA*, KIRAN MANUBHAI PATEL, RAVIKUMAR V. MODI, R. R. BADMANABAN AND DHRUBO JYOTI SEN

SHRI SARVAJANIK PHARMACY COLLEGE, GUJARAT TECHNOLOGICAL UNIVERSITY, ARVIND BAUG, MEHSANA-384001, GUJARAT

virajjatakiya@yahoo.in

ABSTRACT:

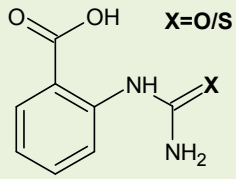
The structure activity relationship study for the synthesized eight compounds has been divided into two series: 2-substituted and 4-substituted phenyl ureas having variable atoms in (X). X=O (urea) and X=S (thiourea) for carboxylic acid and carboxamide substitutions in phenyl ring produces open chain ureas which have been screened for CNS depression study by using closed chain ureas to identify the correlation analogy between closed chain and open chain ureas on CNS depression and sleeping time potentiation. But in the present study we have taken the part of work related to the "Organic reactions monitoring". This approach is there to support and serve as a tool for monitoring the organic reaction synthesis at laboratory level to get a basic and preliminary conclusion for the substitution and linkage by means of electron donating atom of a molecule potential. It may be a useful tool for a researcher and scientist to ascertain the reactions in rapid way.

KEYWORDS: *Electronegativity, DPPH, Antioxidant, Phenyl urea/thiourea (carboxylic acid/carboxamide), IC_{50}*

INTRODUCTION:

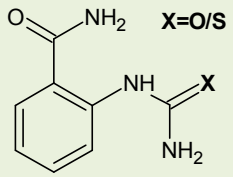
Series of Synthesized Molecules:-

Series: 1



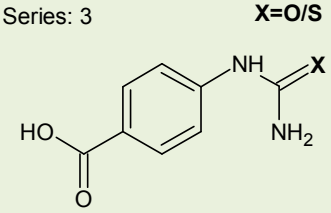
Compound -1: 2-carboxy-phenyl urea
Compound-3: 2-carboxy-phenyl thiourea thiourea

Series: 2

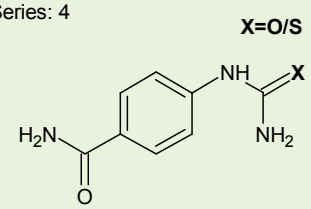


Compound-2: 2-carboxamido-phenyl urea
Compound-4: 2-carboxamido-phenyl

Series: 3



Series: 4



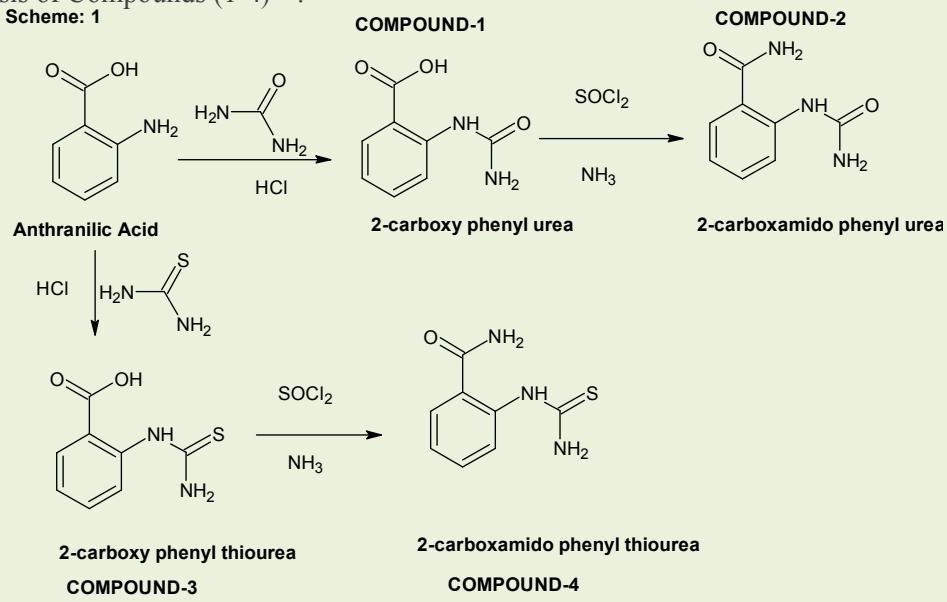
Compound-5: 4-carboxy phenyl urea

Compound-6: 4-carboxamido phenyl urea

Compound-7: 4-carboxy phenyl thiourea thiourea

Compound-8: 4-carboxamido phenyl

Scheme for Synthesis of Compounds (1-4)[1-5]:-



Scheme for Synthesis of Compounds (5-8)[1-5]:-

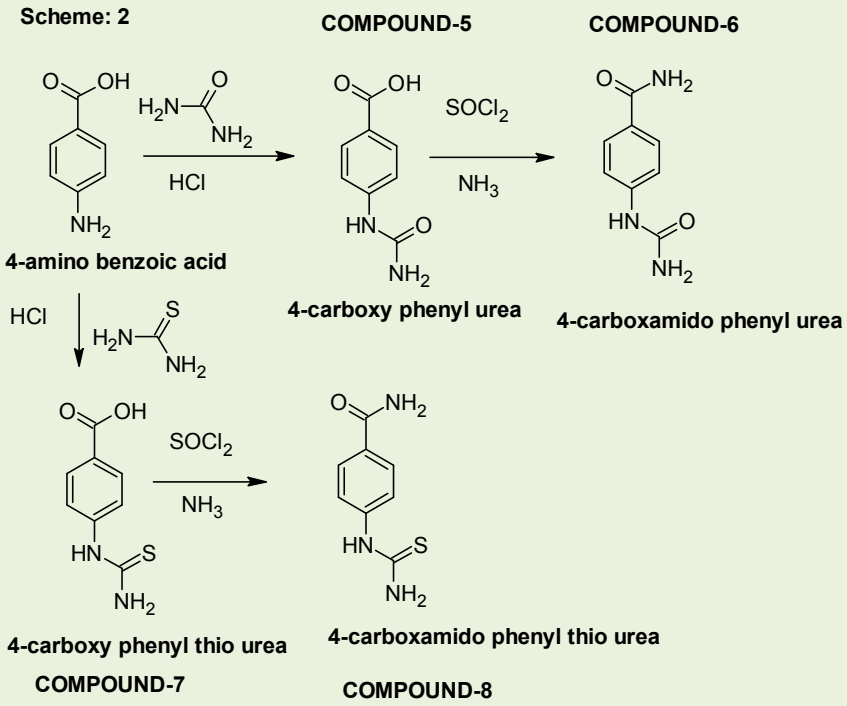


Table-1Solubility Parameters:-

Compounds	Solubility	Polarity
2-carboxy phenyl urea	Hot Water (1mg/ml), Methanol (2mg/ml)	Semi polar
2-carboxamido phenyl urea	Hot Water (1mg/ml)	Semi polar
2-carboxy phenyl thiourea	Water (1mg/ml), Methanol (1mg/ml)	Polar
2-carboxamido phenyl thiourea	Hot Water (1mg/ml)	Semi polar
4-carboxy phenyl urea	Water (1mg/ml), Methanol (2mg/ml)	Polar
4-carboxamido phenyl urea	Hot Water (1mg/ml)	Semi polar
4-carboxy phenyl thiourea	Water (1mg/ml), Methanol	Polar
4-carboxamido phenyl thiourea	Hot Water (1mg/ml)	Semi polar

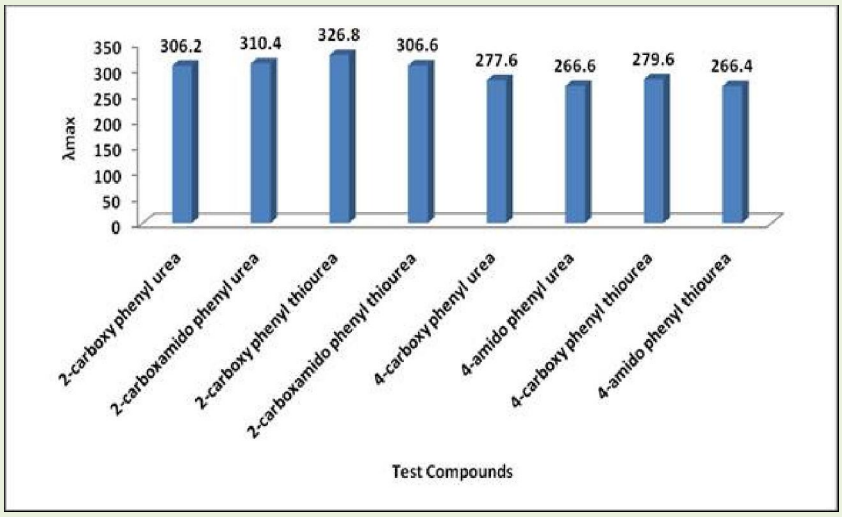
Table-2:U.V. Spectras

Compounds	λ_{max}±SD	Absorbance
2-carboxy phenyl urea	306.2 ±0.97	0.766
2-carboxamido phenyl urea	310.4±0.85	0.960
2-carboxy phenyl thiourea	326.8±0.92	0.939
2-carboxamido phenyl thiourea	306.6±0.71	0.314
4-carboxy phenyl urea	277.6±0.79	0.963
4-carboxamido phenyl urea	266.6±0.82	1.940
4-carboxy phenyl thiourea	279.6±0.90	0.815
4-carboxamido phenyl thiourea	266.4±0.82	1.294

Table-3:Physicochemical Parameters:-

Compounds	% Yield	M.P. °C	Polarity	Molecular Formula	N% Calculated	N% Found
2-CARBOXY PHENYL UREA	77.37%	158-162±1.41	Semi Polar	$C_8H_8N_2O_3$	15.555	15.983
2-CARBOXAMIDO PHENYL UREA	68.33%	101-103±0.5	Semi Polar	$C_8H_9N_2O_2$	16.969	17.218
2-CARBOXY PHENYL THIOUREA	82.48%	176-178±0.56	Polar	$C_8H_8N_2O_2S$	14.285	14.574
2-CARBOXAMIDO PHENYL THIOUREA	56.66%	120-122±0.49	Semi Polar	$C_8H_9N_2OS$	15.469	15.872
4-CARBOXY PHENYL UREA	80.29%	260-262 ± 0.53	Polar	$C_8H_8N_2O_3$	15.555	15.886
4-CARBOXAMIDO PHENYL UREA	48.33%	304-306±0.55	Semi Polar	$C_8H_9N_2O_2$	16.969	17.438
4-CARBOXY PHENYL THIOUREA	76.64%	262-264 ± 0.50	Polar	$C_8H_8N_2O_2S$	14.285	14.674

4-CARBOXAMIDO PHENYL THIOUREA	71.66%	295-297± 0.81	Semi Polar	$C_8H_9N_2OS$	15.469	15.787

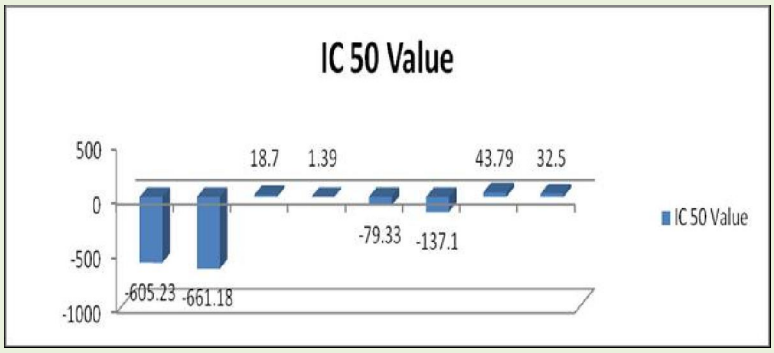


Histogram of Test Compounds

Table-4:IC_{50} values of Test Compounds

Comp's.	Name of the compound	IC 50 Value
1	2-carboxy phenyl urea	- 605.23 ppm
2	2-carboxamido phenyl urea	-661.18 ppm
3	2-carboxy phenyl thio urea	18.7 ppm
4	2-carboxamido phenyl thio urea	1.39 ppm
5	4-carboxy phenyl urea	-79.33ppm
6	4-carboxamido phenyl urea	-137.10 ppm
7	4-carboxy phenyl thio urea	43.79 ppm
8	4-carboxamido phenyl thio urea	32.50 ppm

Table-4:Histogram of IC_{50} of Test Compounds



METHODOLOGY:

DETERMINATION OF RADICAL SCAVANGING POTENTIAL BY AN OXIDANT

Antioxidant assay

The antioxidant activity of synthesised compounds was determined by different *in-vitro* methods such as the DPPH free radical scavenging assay and reducing power methods. The different compounds were dissolved in ethanol at the concentration of 2mg/ml. All the assays were carried out in triplicate and average value was considered.

(a) DPPH Radical scavenging activity

DPPH scavenging activity of the synthesised compounds were carried out according to the method of Koleva *et al* 2002; Mathiesen *et al* 1995. 2 ml of ethyl alcohol solution of synthesised compounds at different concentration (20-100µgml^{-1}) was mixed with 0.8 ml of Tris HCl buffer (100Mm, pH 7.4). One ml DPPH (500 Mm in ethanol) solution was added to above mixture. The mixture was shaken vigorously and incubated for 30min in room temperature. Absorbance of the resulting solution was measured at 517nm UV-Visible Spectrophotometer (Shimadzu 1700, INDIA). All the assays were carried out in triplicates with BHA (Butylated Hydroxy Anisole) as a positive control. Blank was prepared without the addition of DPPH and for control 0.2 ml of ethyl alcohol (without synthesised compounds) was added. Percentage of DPPH scavenging activity determined as follows.

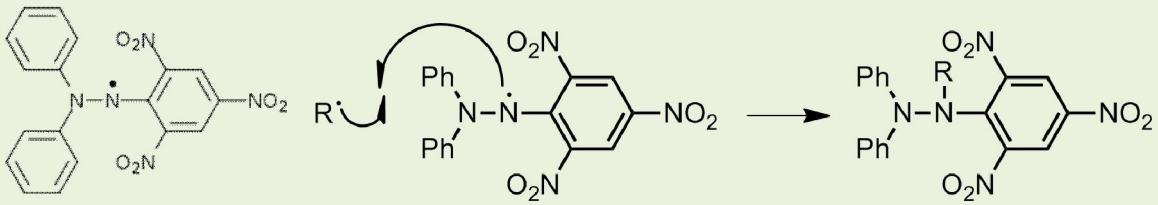
% DPPH radical-scavenging = (Absorbance of control – Absorbnce of test sample) ÷ (Absorbance of control) × 100

Where A=absorbance of the extract and DPPH at 517nm after 30min of reaction

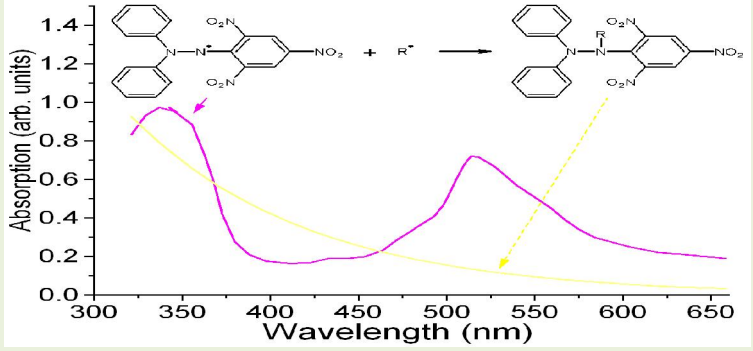
Abs. Cont. =absorbance of DPPH at 517nm as a control, Abs. test =an absorbance of extract at 517nm as a blank

Control was the DPPH solution without synthesised compounds. Purified sample 2 mg/ml in ethyl alcohol of synthesised compounds were taken for antioxidant activity with a standard BHA (Butylated Hydroxy Anisole) antioxidant.

Decreased absorbance of the reaction mixture indicates stronger DPPH radical-scavenging activity.[6-10]



IUPAC Nameof DPPH: di (phenyl)-(2,4,6-trinitrophenyl)iminoazanium

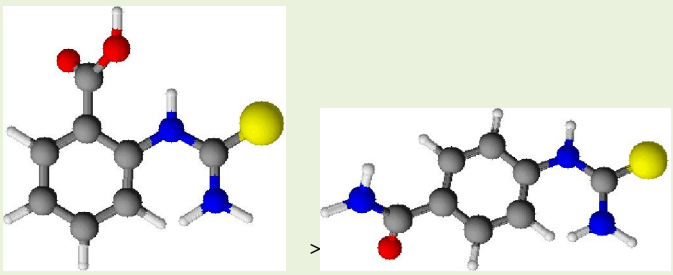


CONCLUSION:

DPPH is a common abbreviation for an organic Chemical Compound 1,1-diphenyl-2-picrylhydrazyl radical. It is a dark-colored crystalline powder composed of stable free-radical molecules. DPPH has two major applications, both in laboratory research: one is a monitor of chemical reactions involving radicals and another is a standard of the position and intensity of electron paramagnetic resonance signals.

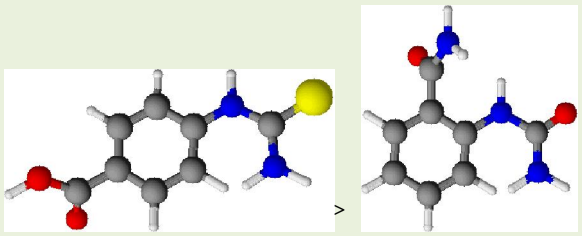
Potentiality of λ_{max} in UV:

2-CARBOXY PHENYL THIOUREA (326.8±0.92)>4-CARBOXAMIDO PHENYL THIOUREA (266.4±0.82)



Potentiality of IC_{50} value:

4-CARBOXY PHENYL THIOUREA (43.79 ppm)>2-CABOXAMIDO PHENYL UREA (-661.18 ppm)



Electronegative atoms O and S both have two lone pair of electrons but electronegativity for O: 3.44>N: 3.04>S: 2.58, so in λ_{max} in UV thiourea compound gives maximum absorption but in –COOH the electronegativity is more than $–CONH_2$. Same thing takes place during IC_{50} value.

This approach is there to support and serve as a tool for monitoring the organic reaction synthesis at laboratory level to get a basic and preliminary conclusion for the substitution and linkage by means of electron donating atom of a molecule potential. It may be a useful tool for a researcher and scientist to ascertain the reactions in rapid way.

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