Research Journal of Pharmaceutical Sciences ISSN 2319 – 555X
Vol. **14(2),** 27-37, December **(2025)** *Res. J. Pharmaceutical Sci.*

Determination of Paracetamol from different Medicinal sample Colorimetrically and its FTIR study

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Available online at: www.isca.in, www.isca.me
Received 11th August 2025, revised 17th October 2025, accepted 28th November 2025

Abstract

Paracetamol (acetaminophen or para-hydroxyacetanilide) is a non-opioid analgesic and antipyretic agent used to treat fever and mild to moderate pain. Paracetamol is available in oral, suppository, and intravenous forms. In some formulations, paracetamol is combined with the opiate codeine, sometimes referred to as co-codamol (BAN) and Panadeine. Paracetamol is also combined with other opioids such as dihydrocodeine, referred to as co-dydramol (British Approved Name (BAN)), oxycodone or hydrocodone. Another very commonly used analgesic combination includes paracetamol in combination with propoxyphene napsylate. A combination of paracetamol, codeine, and the doxylamine succinate is also available. Paracetamol is sometimes combined with phenylephrine hydrochloride. Sometimes a third active ingredient, such as ascorbic acid, caffeine, chlorpheniramine maleate, or guaifenesin is added to this combination. Present study deals with determination of paracetamol from different medicinal sample Macfast 500, SumoL 650, LANOL ER 650, P-250, Arden 650, Calpal 500, Polo-650, Wellpar 500, MEDOMOL 650 and Dolo 650 Colorimetrically and FTIR study of Dolo 650Paracetamol medicinal sample. Determination of paracetamol from different medicinal sample colorimetrically involves preparation of standard paracetamol solution, paracetamol medicinal sample solution and resorcinol solution. Different systems were prepared. Absorbance of standard paracetamol system was taken at different wavelength. And λmax was calculated. Which was 530 nm. Absorbance of standard paracetamol systems were taken at 530 nm and Calibration plot was prepared. Then absorbance of paracetamol medicinal systems were taken at 530 nm and concentration of paracetamol present in different medicinal sample were calculated from calibration plot. Paracetamol with resorcinol gave azodye and the concentration of paracetamol was investigated colorimetrically. The results thus obtained are in good agreement with the quoted values. The method is simple, rapid and precise. FTIR spectra of Dolo 650 Paracetamol medicinal sample is obtained at room temperature by using an FTIR Spectrophotometer – Perkin Elmer – Spectrum RX-IFTIR. The spectra is collected in a range from 400 to 4000 cm^{-1}. Interpretation of FTIR spectra of Paracetamol shows various groups such as C-H bending - Aromatic Compound, C-N stretching - Amine, O-H stretching - alcohol, O-H bending - Alcohol, O-H bending - Phenol, N-H stretching - Secondary amine, S-H stretching - Thiol, C=C stretching - Conjugated alkene, C≡C stretching - Alkyne, C≡N stretching – Nitrile.

Keywords: Paracetamol, Macfast 500, SumoL 650, LANOL ER 650, P-250, Arden 650, Calpal 500, Polo-650, Wellpar 500, MEDOMOL 650 and Dolo 650, Colorimetrically and FTIR study.

Introduction

Paracetamol (acetaminophen or para-hydroxyacetanilide) is a non-opioid analgesic and antipyretic agent used to treat fever and mild to moderate pain[1-3]. Paracetamol is available in oral, suppository, and intravenous forms[4]. In some formulations, paracetamol is combined with the opiate codeine, sometimes referred to as co-codamol (BAN) and Panadeine in Australia. As of 1 February 2018, medications containing codeine also became prescription-only in Australia[5].

Paracetamol is sometimes combined with phenylephrine hydrochloride. Sometimes a third active ingredient, such as ascorbic acid, caffeine, chlorpheniramine maleate, or guaifenesinis added to this combination[6-9].

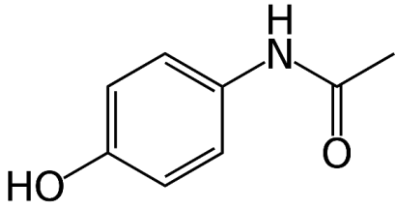


Figure-1: Paracetamol, $C_8H_9NO_2$.

A spectrophotometric method is proposed for the determination of paracetamol in pure form and in tablets. The method depends on reaction of the drug with ammonium molybdate in strongly acidic medium to produce molybdenum blue. Effects of variables such as temperature, heating time, acidity and reagent concentration have been evaluated to permit selection of the most advantageous technique[10].

A simple and rapid spectrophotomitric method has been developed to determine orphenadrine citrate and paracetamol in tablet formulations. The first step was based on the reaction of orphenadrine citrate with 1-amino naphthalene and sodium nitrite with heating for 6 min at 50^{O}C to give an orange colour having a maximum absorbance at 462 nm. The optimization of the reaction conditions is investigated. In the second step, paracetamol was analyzed after solving it in distilled water by taking the first order derivative spectroscopy and that to eliminate spectral interference with orphenadrine citrate[11].

A rapid and simple spectrophotometric method is reported for the determination of paracetamol in a commercially available tablet formulation. The method is based on the diazotization of hydrolyzed paracetamol with 8-hydroxyquinoline as a coupler to form stable azo dyes color solution. The concentration of drug paracetamol was investigated by spectrophotometrically. The method reported may be used to determine the trace amount of paracetamol in any clinical samples with accuracy and precision[12].

The UV spectrophotometric methods for simultaneous quantitative determination of paracetamol and tramadol in paracetamol-tramadol tablets were developed. The spectrophotometric data obtained were processed by means of partial least squares (PLS) and genetic algorithm coupled with PLS (GA-PLS) methods in order to determine the content of active substances in the tablets. The results gained by chemometric processing of the spectroscopic data were statistically compared with those obtained by means of validated ultra-high performance liquid chromatographic (UHPLC) method[13].

The synthesis of magnetic iron oxide/reduced graphene oxide (Fe_3O_4/rGO) and its application to the electrochemical determination of paracetamol using Fe_3O_4/rGO modified electrode were demonstrated.

The obtained materials were characterized by means of X-ray diffraction (XRD), nitrogen adsorption/desorption isotherms, X-ray photoelectron spectroscopy (XPS), transmission electron microscope (TEM), Fourier transform infrared spectroscopy (FTIR), and magnetic measurement[14].

Comtrex tablets composed of paracetamol, pseudoephedrine and brompheniramine are widely used for relieving symptoms related to common cold. Study has overcome the challenging dosage form ratio (250:15:1) and proposed chromatographic methods for analyzing the ternary combination were utilized displaying different apparatus, solvents and sensitivity ranges.

Three chromatographic methods namely thin layer chromatography (TLC), high performance liquid chromatography with ultra-violet detection (HPLC–UV) and ultra-performance liquid chromatography coupled to tandem mass spectrometry (UPLC-MS/MS) were developed and validated for the simultaneous determination of the three drugs[15].

It is described the use of 1,3 dinitrobenzene or 2,4 dinitrophenyl hydrazine is used as coupling agent for the spectrophotometric determination of paracetamol. This method is easy and simple based on the reaction of acid hydrolysis of paracetamol to produce p-aminophenol, which in turn reacts with nitrite in acidic standard to form diazonium ion, which is coupled with coupling agent in basic standard to produce azo dyes. The optimal reaction circumstances and other analytical parameters are evaluated. Interference due to foreign organic compounds has been studied. The method has been effectively applied to the determination of paracetamol in pharmaceutical samples[16].

A new application for microcomposites based on carbon paste (CP) and La_2O_3 (LaOX) has been presented. This simple and versatile microcomposite (LaOX/CPE) was applied toward the determination of paracetamol (PCM) through proton oxidation by square wave adsorptive voltammetry (SWAdV). The accuracy of the new method was evaluated with tap water spiked with known quantities of PCM, while ascorbic acid, caffeine, and acetylsalicylic acid were used for interference studies. The usefulness of the microcomposite was shown to have acceptable results when applied to detect and quantify PCM in various forms of a pharmaceutical dose, such as solid tablets, fruit-flavored powders for colds and syrups for children[17].

Present study deals with determination of paracetamol from different medicinal sample Macfast 500, SumoL 650, LANOL ER 650, P-250, Arden 650, Calpal 500, Polo-650, Wellpar 500, MEDOMOL 650 and Dolo 650 Colorimetrically and FTIR study of Dolo 650 Paracetamol medicinal sample.

Methodology

Preparation of Standard Paracetamol Solution: 0.5 g Paracetamol was taken to it 10 ml glacial acetic acid and 90 ml distilled water was added.

Preparation of sample Solution: 1 Tablet of different Paracetamol medicinal sample such as Dolo 650, Macfast 500, SumoL 650, LANOL ER 650 MG, P-250, Arden 650, Calpal 500 mg, Polo-650, Wellpar 500, MEDOMOL 650 were taken to it 10 ml glacial acetic acid and 90 ml distilled water was added.

Preparation of Resorcinol solution: 0.1 g of Resorcinol was dissolved in 100 ml distilled water. Different systems were prepared as follows.

Table-1: Preparation of Paracetamol medicinal sample Solution.

System No.	Paracetamol Solution, ml	Resorcinol Solution, ml	Distilled Water, ml
Standard Paracetamol Solution			
1	1	1	8
2	2	1	7
3	3	1	6
4	4	1	5
5	5	1	4
Mac Fast 500			
6	1	1	8
7	2	1	7
8	3	1	6
9	4	1	5
10	5	1	4
Sumol 650			
11	1	1	8
12	2	1	7
13	3	1	6
14	4	1	5
15	5	1	4
LANOL ER 650			
16	1	1	8
17	2	1	7
18	3	1	6
19	4	1	5
20	5	1	4
P- 250			
21	1	1	8
22	2	1	7
23	3	1	6
24	4	1	5
25	5	1	4
Arden 650			
26	1	1	8
27	2	1	7
28	3	1	6
29	4	1	5
30	5	1	4
Calpal 500			
31	1	1	8
32	2	1	7
33	3	1	6
34	4	1	5
35	5	1	4
Polo 500			
36	1	1	8
37	2	1	7
38	3	1	6
39	4	1	5
40	5	1	4
Wellpar 500			
41	1	1	8
42	2	1	7
43	3	1	6
44	4	1	5
45	5	1	4
MEDOMOL 650			
46	1	1	8
47	2	1	7
48	3	1	6
49	4	1	5
50	5	1	4
Dolo 650			
51	1	1	8
52	2	1	7
53	3	1	6
54	4	1	5
55	5	1	4

Absorbance of System No. 1 was taken at different wavelength. And λmax was calculated. Which was 530 nm. Absorbance of System No. 1 to 5 was taken at 530 nm and Calibration plot was prepared. Then absorbance of System No. 6 to 55 was taken at 530 nm and concentration of paracetamol present in different medicinal sample were calculated from calibration plot.

FTIR Study of Dolo 650 Paracetamol medicinal sample: FTIR can be routinely used to identify the functional groups and identification/quality control of raw material/finished products. Spectrum RX-I offers fast throughput and rapid access to reliable and dependable IR results. High signal to noise ratio makes FTIR more useful for difficult samples. It has resolution of 1 cm1 and scan range of 4000 cm-1 to 250 cm-1. In the normal mode around 10 mg sample is required in the form of fine powder. The sample can be analyzed in the form of liquid, solid and thin films also.

FTIR spectra of Dolo 650 Paracetamol medicinal sample is obtained at room temperature by using an FTIR Spectrophotometer – Perkin Elmer – Spectrum RX-IFTIR. The spectra is collected in a range from 400 to 4000 cm^{-1}

Table-2: Absobance of System No. 1 at different wavelength.

Filter	Absorbance
400	0.01
420	0.02
470	0.03
500	0.04
530	0.05
620	0.04
660	0.03
700	0.03

Table-3: Absorbance of Different systems at λmax 530 nm.

System No.	Absorbance
Standard Paracetamol Solution	
1	0.07
2	0.13
3	0.20
4	0.26
5	0.33
Macfast 500	
6	0.06
7	0.12
8	0.19
9	0.24
10	0.29
SumoL 650	
11	0.08
12	0.16
13	0.24
14	0.33
15	0.40
LANOL ER 650	
16	0.09
17	0.15
18	0.24
19	0.34
20	0.39
P-250	
21	0.03
22	0.07
23	0.10
24	0.11
25	0.16
Arden 650	
26	0.08
27	0.15
28	0.24

29	0.34
30	0.40
Calpal 500	
31	0.07
32	0.12
33	0.20
34	0.25
35	0.29
Polo-650	
36	0.09
37	0.17
38	0.25
39	0.33
40	0.41
Wellpar 500	
41	0.06

42	0.13
43	0.19
44	0.25
45	0.28
MEDOMOL 650	
46	0.08
47	0.17
48	0.25
49	0.34
50	0.41
Dolo 650	
51	0.09
52	0.18
53	0.24
54	0.35
55	0.42

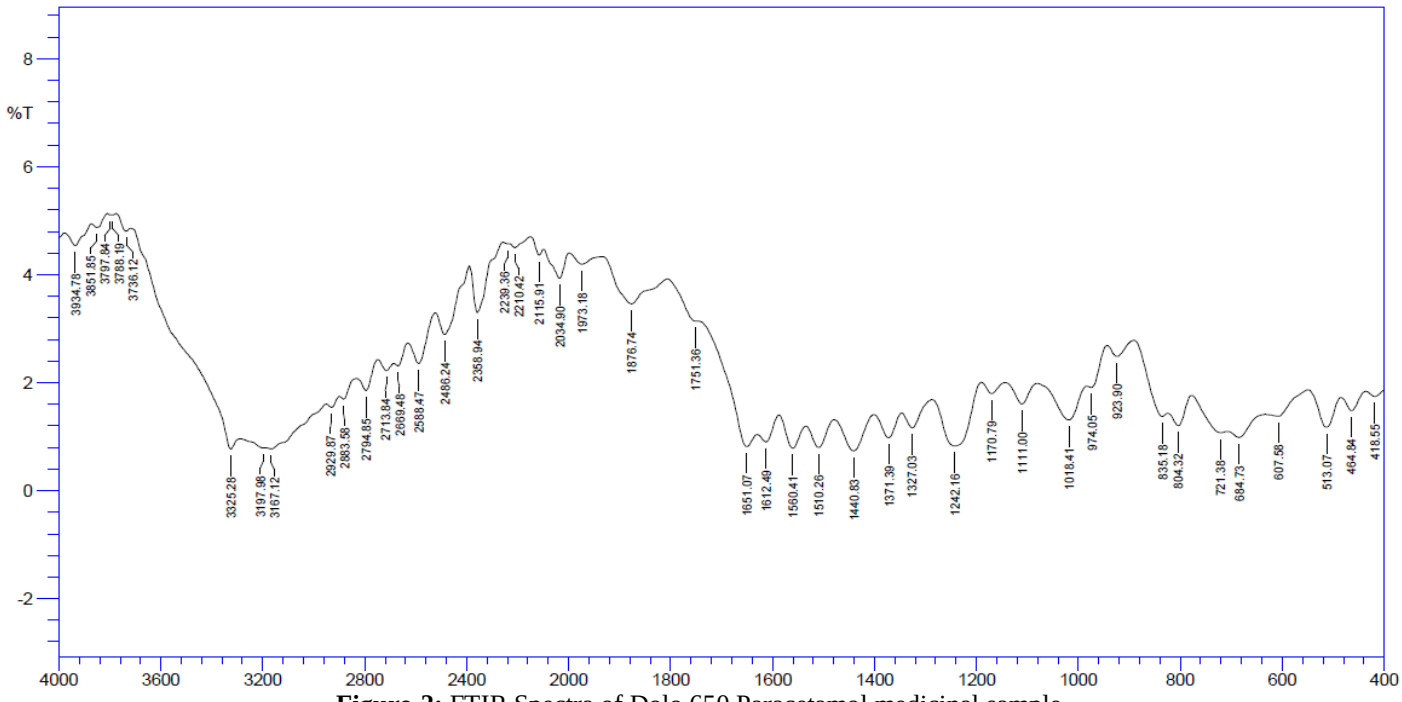


Figure-2: FTIR Spectra of Dolo 650 Paracetamol medicinal sample.

Results and Discussion

Table-4: Measurement of Absorbance of different medicinal sample Colorimetrically.

System No	Volume of Standard paracetamol solution taken, ml	Amount of paracetamol present, g	Absorbance
1	1	1 x 0.5 / 100 = 0.005	0.07
2	2	2 x 0.5 / 100 = 0.010	0.13
3	3	3 x 0.5 / 100 = 0.015	0.20
4	4	4 x 0.5 / 100 = 0.02	0.26
5	5	5 x 0.5 / 100 = 0.025	0.33

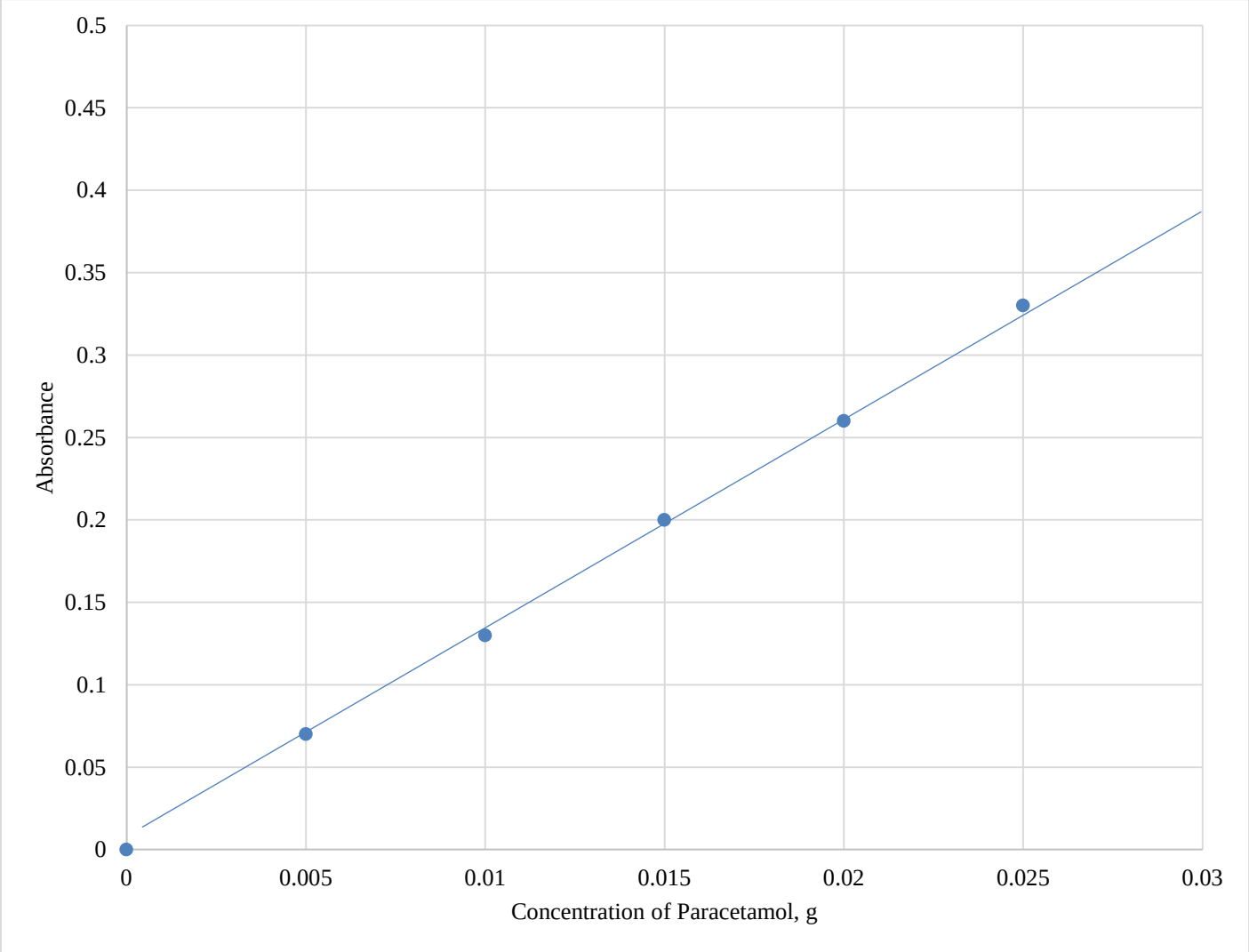


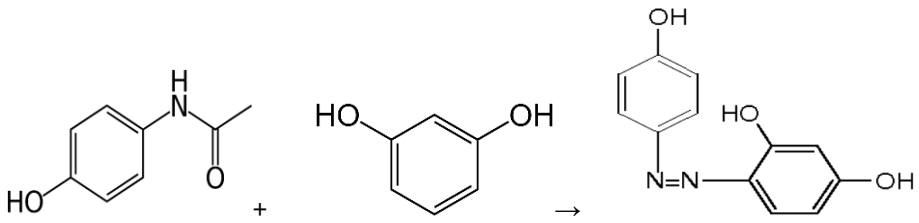
Figure-3: Calibration Plot.

Table-5: Determination of paracetamol from different medicinal sample Colorimetrically.

System No.	Absorbance	Volume of Paracetamol Solution taken, ml	Concentration from Graph, g	Amount of paracetamol per 100 ml, g	Average Amount of paracetamol per 100 ml, g
Macfast 500					
6	0.06	1	0.0046	100 x 0.0046 /1= 0.46	0.4334
7	0.12	2	0.0094	100 x 0.0094 /2=0.47	
8	0.19	3	0.0146	100x0.0146/3= 0.4867	
9	0.24	4	0.0184	100 x 0.0184 / 4= 0.46	
10	0.29	5	0.0220	100 x 0.0220 /5= 0.44	
SumoL 650					
11	0.08	1	0.006	100 x 0.006/ 1 = 0.600	0.6133
12	0.16	2	0.0124	100 x 0.0124/ 2 =0.62	
13	0.24	3	0.0184	100 x 0.0184/ 3= 0.6133	
14	0.33	4	0.025	100 x 0.025 / 4 = 0.625	
15	0.40	5	0.0304	100 x 0.0304 /5= 0.608	
LANOL ER 650					
16	0.09	1	0.007	100 x 0.007 /1 = 0.7	0,6251
17	0.15	2	0.0114	100 x 0.0114/ 2 = 0.57	
18	0.24	3	0.0184	100 x 0.0184 / 3 = 0.6133	
19	0.34	4	0.026	100 x 0.026 /4 = 0.65	
20	0.39	5	0.0296	100 x 0.0296/5 = 0.592	
P-250					
21	0.03	1	0.0024	100 x 0.0024/ 1= 0.24	0.2515
22	0.07	2	0.0054	100 x 0.0054 / 2= 0.27	
23	0.10	3	0.0076	100 x 0.0076 / 3= 0.2533	
24	0.11	4	0.010	100 x 0.010 /4= 0.25	
25	0.16	5	0.0122	100 x 0.0122 /5 = 0.244	
Arden 650					
26	0.08	1	0.006	100 x 0.006 /1= 0.6	0.6083
27	0.15	2	0.0114	100 x 0.0114 / 2= 0.57	
28	0.24	3	0.0184	100 x 0.0184 / 3= 0.6133	
29	0.34	4	0.026	100 x 0.026 /4= 0.65	

30	0.40	5	0.0304	100 x 0.0304 /5= 0.608	
Calpal 500					
31	0.07	1	0.0054	100 x 0.0054 /1=0.54	0.485
32	0.12	2	0.0094	100 x 0.0094 / 2= 0.47	
33	0.20	3	0.015	100 x 0.015 / 3= 0.5	
34	0.25	4	0.019	100 x 0.019 /4= 0.475	
35	0.29	5	0.022	100 x 0.022/5= 0.44	
Polo-650					
36	0.09	1	0.007	100 x 0.007 /1= 0.7	0.6457
37	0.17	2	0.013	100 x 0.013 / 2 = 0.65	
38	0.25	3	0.019	100 x 0.019/ 3 =0.6333	
39	0.33	4	0.025	100 x 0.025 /4 = 0.625	
40	0.41	5	0.031	100 x 0.031/5 = 0.62	
Wellpar 500					
41	0.06	1	0.0046	100 x 0.0046 /1 = 0.46	0.4699
42	0.13	2	0.010	100 x 0.010/ 2 = 0.5	
43	0.19	3	0.0146	100 x 0.0146 / 3 = 0.4867	
44	0.25	4	0.019	100 x 0.019 /4 = 0.475	
45	0.28	5	0.0214	100 x 0.0214 /5 = 0.428	
MEDOMOL 650					
46	0.08	1	0.006	100 x 0.006 /1 = 0.6	0.6307
47	0.17	2	0.013	100 x 0.013/ 2 = 0.65	
48	0.25	3	0.019	100 x 0.019 / 3 = 0.6333	
49	0.34	4	0.026	100 x 0.026 /4 = 0.65	
50	0.41	5	0.031	100 x 0.031 /5 = 0.62	
Dolo 650					
51	0.09	1	0.007	100 x 0.007 /1 = 0.7	0.6559
52	0.18	2	0.0138	100 x 0.0138 / 2 = 0.69	
53	0.24	3	0.0184	100 x 0.0184 / 3 = 0.6133	
54	0.35	4	0.0256	100 x 0.0256 / 4 = 0.64	
55	0.42	5	0.0318	100 x 0.0318 /5 = 0.636	

Paracetamol with resorcinol gave azodye and the concentration of paracetamol was investigated colorimetrically. The results thus obtained are in good agreement with the quoted values. The method is simple, rapid and precise.



Paracetamol Resorcinol Azo Dye

Figure-4: Reaction of Paracetamol with resorcinol giving azo dye.

Table-6: FTIR Analysis of Dolo 650 Paracetamol medicinal sample.

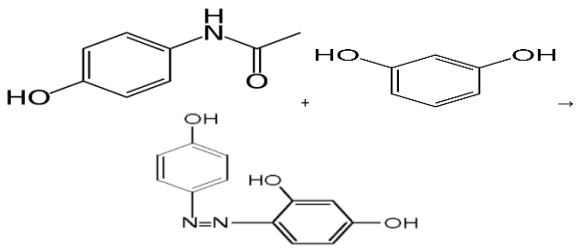
Spectral region wave number cm^{-1}	Pattern and intensity of Band	Bond causing Absorption	Compound Class
3934.78	Low and Broad	-	-
3851.85	Low and Broad	-	-
3797.84	Low and Broad	-	-
3788.19	Low and Broad	-	-
3736.12	Low and Broad	-	-
3325.28	Strong and Broad	N-H stretching	Secondary amine
3197.98	Strong and Broad	O-H stretching	alcohol
3167.12	Strong and Broad	O-H stretching	alcohol
2929.87	Low and Broad	-	-
2883.58	Low and Broad	-	-
2794.85	Low and Broad	-	-
2713.84	Low and Broad	-	-
2669.48	Low and Broad	-	-
2588.47	Low and Broad	S-H stretching	Thiol
2486.24	Low and Broad	-	-
2358.94	Moderate and Sharp	-	-
2239.36	Low and Broad	C≡N stretching	Nitrile
2210.42	Low and Broad	C≡C stretching	Alkyne
2115.91	Low and Broad	C≡C stretching	Alkyne
2034.90	Low and Broad	-	-
1973.18	Low and Broad	C-H bending	Aromatic Compound
1876.74	Moderate and Broad	C-H bending	Aromatic Compound

1751.36	Low and Broad	-	-
1651.07	Moderate and Broad	C=C stretching	Conjugated alkene
1612.49	Moderate and Broad	C=C stretching	Conjugated alkene
1560.41	Low and Broad	-	-
1510.26	Low and Broad	-	-
1440.83	Low and Broad	-	-
1371.39	Moderate and Broad	O-H bending	Alcohol
1327.03	Moderate and Broad	O-H bending	Phenol
1242.16	Moderate and Broad	C-N stretching	Amine
1170.79	Low and Broad	-	-
1111.00	Low and Broad	-	-
1018.41	Moderate and Broad	-	-
974.05	Low and Broad	-	-
923.90	Low and Broad	-	-
835.18	Moderate and Broad	-	-
804.32	Moderate and Broad	-	-
721.38	Low and Broad	-	-
684.73	Low and Broad	-	-
607.58	Low and Broad	-	-
513.07	Moderate and Broad	-	-
464.84	Low and Broad	-	-
418.55	Low and Broad	-	-

Interpretation of FTIR spectra of Paracetamol shows various groups such as C-H bending - Aromatic Compound, C-N stretching - Amine, O-H stretching - alcohol, O-H bending - Alcohol, O-H bending - Phenol, N-H stretching - Secondary amine, S-H stretching - Thiol, C=C stretching - Conjugated alkene, C≡C stretching - Alkyne, C≡N stretching – Nitrile.

Conclusion

Paracetamol with resorcinol gave azodye and the concentration of paracetamol was investigated colorimetrically. The results thus obtained are in good agreement with the quoted values. The method is simple, rapid and precise.



Paracetamol **Resorcinol Azo Dye**

Figure-5: Reaction of Paracetamol with resorcinol giving azodye.

FTIR Analysis of Dolo 650 Paracetamol medicinal sample: Interpretation of FTIR spectra of Paracetamol shows various groups such as C-H bending - Aromatic Compound, C-N stretching - Amine, O-H stretching - alcohol, O-H bending - Alcohol, O-H bending - Phenol, N-H stretching - Secondary amine, S-H stretching - Thiol, C=C stretching - Conjugated alkene, C≡C stretching - Alkyne, C≡N stretching – Nitrile.

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