

## RESEARCH ARTICLE

## Polarographic determination of *Paracetamol* by calibration method using acetic acid and different maxima suppressors

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Manuscript Details	ABSTRACT
<p>Received : 28.06.2015 Revised :15.07.2015 Revised received : 09.09.2015 Accepted: 21.09.2015 Published: 05.10.2015</p> <p><b>ISSN: 2322-0015</b></p> <p><b>Editor: Dr. Arvind Chavhan</b></p> <p><b>Cite this article as:</b> Gupta Swaroopa Rani N. Polarographic determination of <i>Paracetamol</i> by calibration method using acetic acid and different maxima suppressors, <i>Int. Res. J. of Science &amp; Engineering</i>, 2015; Vol. 3 (5):214-223.</p> <p><b>Copyright:</b> © Author(s), This is an open access article under the terms of the Creative Commons Attribution Non-Commercial No Derivs License, which permits use and distribution in any medium, provided the original work is properly cited, the use is non-commercial and no modifications or adaptations are made.</p>	<p>Paracetamol is a common analgesic and antipyretic drug. Their determination in pharmaceuticals is of paramount importance, since an overdose of paracetamol can cause toxic effects. The aim of the present study is to do polarographic determination of paracetamol by calibration method using acetic acid and different maxima suppressors such as gelatin, fuchsin, methyl red, thymol blue and bromocresol green. Calibration method has been developed and applied for the determination of paracetamol present in some synthetic as well as medicinal samples using selected maxima suppressor-supporting electrolyte system. The method is strictly empirical, and no assumptions, except correspondence with the conditions of the calibration are made. According to the Ilkovic equation, with all other factors constant, <math>i_d = kC</math>. Where k is a constant defined by Ilkovic equation. This relation is the foundation of quantitative polarographic analysis and its general validity is well established. The results of polarographic determination of paracetamol from synthetic and medicinal samples by calibration method are in good agreement with the quoted values.</p> <p><b>Keywords:</b> Paracetamol, Acetic Acid, Gelatin, Fuchsin, Methyl Red, Thymol Blue, Bromocresol Green</p> <p><b>INTRODUCTION</b></p> <p>A flow injection- spectrophotometric determination of paracetamol, the influence of foreign species and the determination of paracetamol in several pharmaceutical formulations were reported by Calatayud <i>et al.</i> (1986). A simple, rapid and accurate</p>

method for the simultaneous determination of ascorbic acid, caffeine and paracetamol in drug formulations has been developed and results are reported for several commercially available drugs Lau *et al.* (1989). A flow-injection spectrofluorimetric determination of paracetamol, the influence of foreign species and the determination of paracetamol in several pharmaceutical formulations are also reported (Calatayud and Benito, 1990). A simple, but sensitive, micro-assay for paracetamol in blood and plasma is described and the method was applied to a study of gastric emptying in patients before and after cardiac surgery (Whelpton *et al.*, 1993). A polarographic procedure was described for the determination of paracetamol and salicylamide after treatment with nitrous acid and different experimental parameters affecting the derivatization process and the polarographic analysis were studied and the procedure was applied to the analysis of some pharmaceutical dosage forms (Walash *et al.*, 1994). A simple and fast analytical procedure was proposed for the simultaneous determination of paracetamol, acetylsalicylic acid and caffeine in pharmaceuticals by means the partial least square treatment of the spectrophotometric absorbance data between 216 and 300 nm, taken at 5 nm intervals (Bouhsain *et al.*, 1997). After a large drug scanning, the system Luminol-H<sub>2</sub>O<sub>2</sub>-Fe(CN)<sub>6</sub><sup>3-</sup> was proposed for first time for the indirect determination of paracetamol and The influence of foreign compounds was studied and, the method was applied to determination of the drug in three different pharmaceutical formulations (Alapont *et al.*, 1999). A voltammetric method, aided by chemometrics, was developed for the simultaneous determination of paracetamol and phenobarbital in pharmaceuticals and the proposed method was verified by an established HPLC method, and its practical application was demonstrated with the determination of paracetamol and phenobarbital in several commercial tablets with satisfactory results (Ni *et al.*, 2004). Bosch *et al* evaluated the utility of different techniques for quantification of paracetamol content in pharmaceutical formulations and biological samples (Bosch *et al.*, 2006). A novel type of carbon-coated nickel magnetic nanoparticles modified glass carbon electrodes (C-

Ni/GCE) was fabricated and the electrochemical properties of paracetamol were studied on the C-Ni/GCE and has been applied to the determination of paracetamol in effervescent dosage samples (Wang *et al.*, 2007). Effect of supporting electrolytes and maxima suppressors on polarographic anodic waves of paracetamol was done (Gupta, 2015; 2014a; 2014b) so that these data can be utilized for development of procedures for their quantitative estimations and applications to various pharmaceutical preparations. Polarographic determination of paracetamol in pharmaceutical preparations using 0.008% gelatin as maxima suppressor and 0.1 M HClO<sub>4</sub> as supporting electrolyte by calibration as well as internal standard addition method was done (Gupta, 2015; 2014c).

Paracetamol is a common analgesic and antipyretic drug that is used for the relief of fever, headaches and other minor aches and pains. Their determination in pharmaceuticals is of paramount importance, since an overdose of paracetamol can cause fulminating hepatic necrosis and other toxic effects. The aim of the present study is to do polarographic determination of paracetamol by calibration method using acetic acid and different maxima suppressors such as gelatin, fuchsin, methyl red, thymol blue and bromocresol green.

## **MATERIAL AND METHOD**

Standard solutions of different concentrations of the paracetamol were prepared, under different experimental conditions of maxima suppressor-supporting electrolyte combination as given in Table 1. Similar solutions were prepared for medicinal samples. 50 ml total volume was maintained for each measurement. Polarograms of all system were recorded on D.C. Recording Polarograph using Omniscribe recorder between 200 to 1300 mV using Rotating Platinum micro Electrode (RPE) as anode and Saturated Calomel Electrode (S.C.E.) as cathode. The heights of the waves obtained were measured and plotted as a function of the concentration.

**Table 1: Optimum concentration of Maxima Suppressor-Supporting Electrolyte for Paracetamol determination.**

Maxima Suppressor	Supporting electrolyte
1 x 10 <sup>-6</sup> % Gelatin	0.1 M CH <sub>3</sub> COOH
5 x 10 <sup>-5</sup> % Fuchsin	0.1 M CH <sub>3</sub> COOH
1 x 10 <sup>-3</sup> % Methyl red	0.1 M CH <sub>3</sub> COOH
1.5 x 10 <sup>-2</sup> % Thymol blue	2.0 M CH <sub>3</sub> COOH
2.5 x 10 <sup>-2</sup> % Bromocresol green	1.0 M CH <sub>3</sub> COOH

**RESULTS AND DISCUSSION**

Paracetamol from six categories of drugs were analyzed by polarographic calibration method and they are Analgesics and antipyretics, Sedatives and tranquillisers, Vasoconstrictors and migraine treatments, Non-steroid anti-inflammatory drugs, Muscle relaxants, Expectorants, cough-suppressants, mucolytics and decongestants. Current vs applied potential graph of different amount of paracetamol using acetic acid and different maxima suppressors such as gelatin, fuchsin, methyl red, thymol blue and bromocresol green are shown in Fig. 1 to 5.

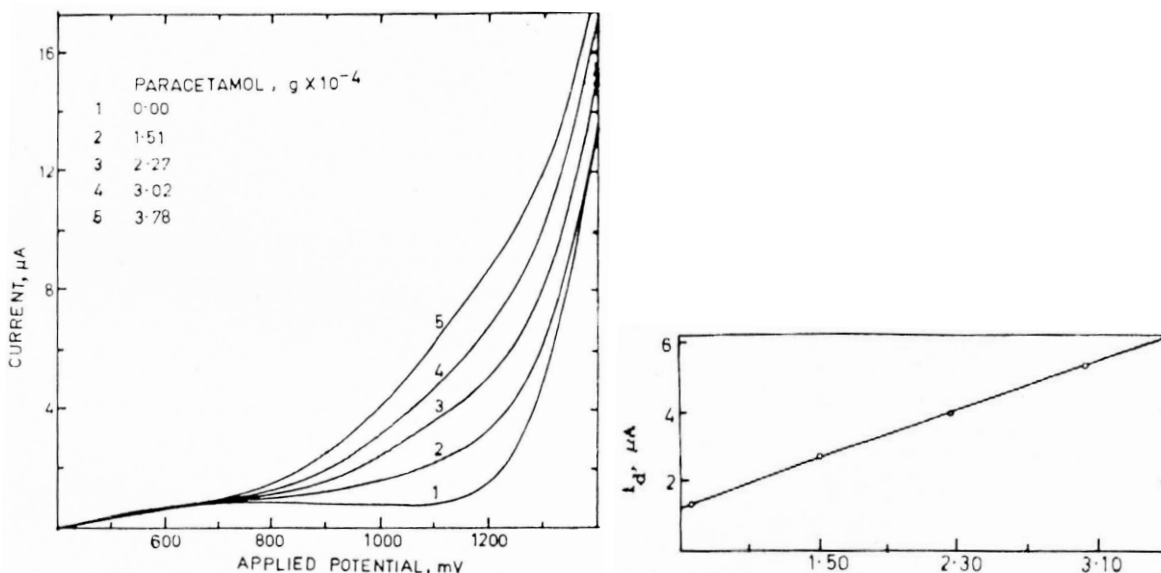


Fig. 1. (a) Calibration polarogram for Paracetamol determination in 0.1 M CH<sub>3</sub>COOH with 1 x 10<sup>-6</sup> % Gelatin; (b) Calibration curve for Paracetamol

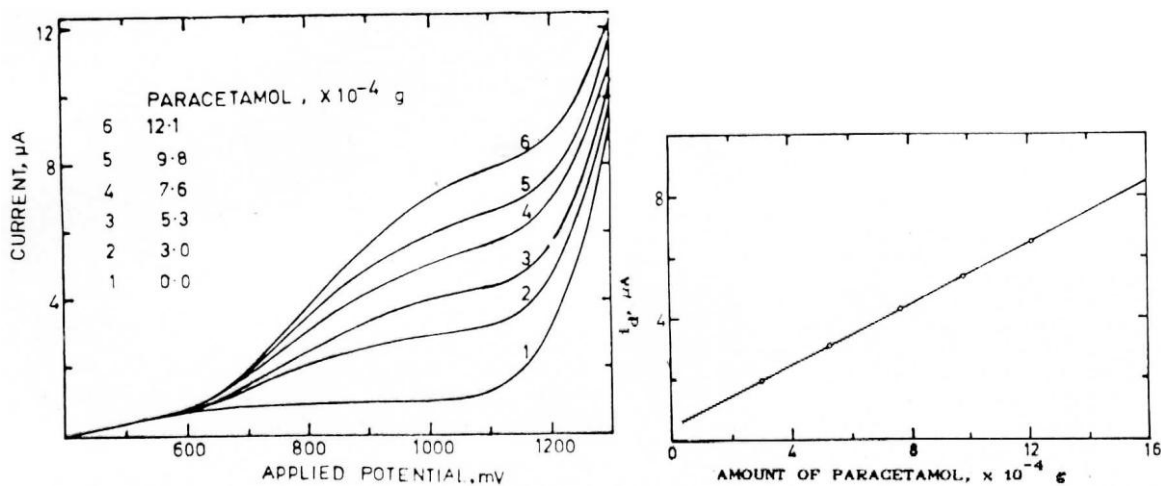


Fig. 2. (a) Calibration polarogram for Paracetamol determination in 0.1 M CH<sub>3</sub>COOH with 5 x 10<sup>-5</sup> % Fuchsin.; (b) Calibration curve for Paracetamol

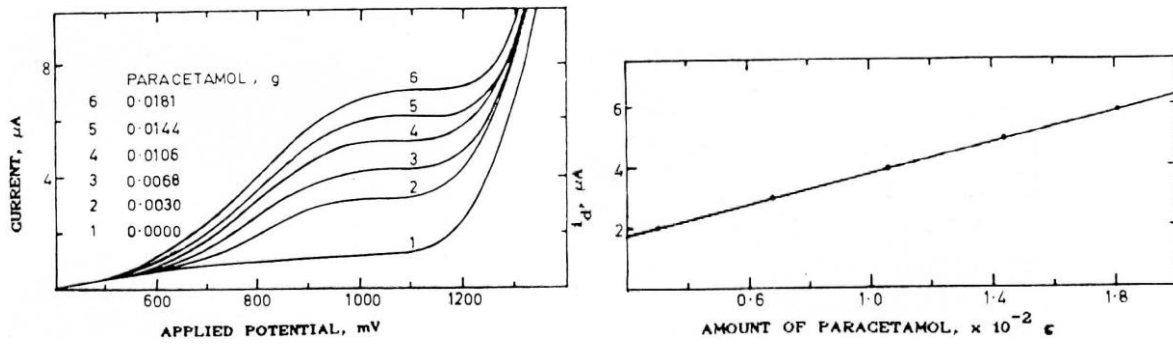


Fig. 3. (a) Calibration polarogram for Paracetamol determination in 0.1 M CH<sub>3</sub>COOH with 1 x 10<sup>-3</sup> % Methyl red; (b) Calibration curve for Paracetamol

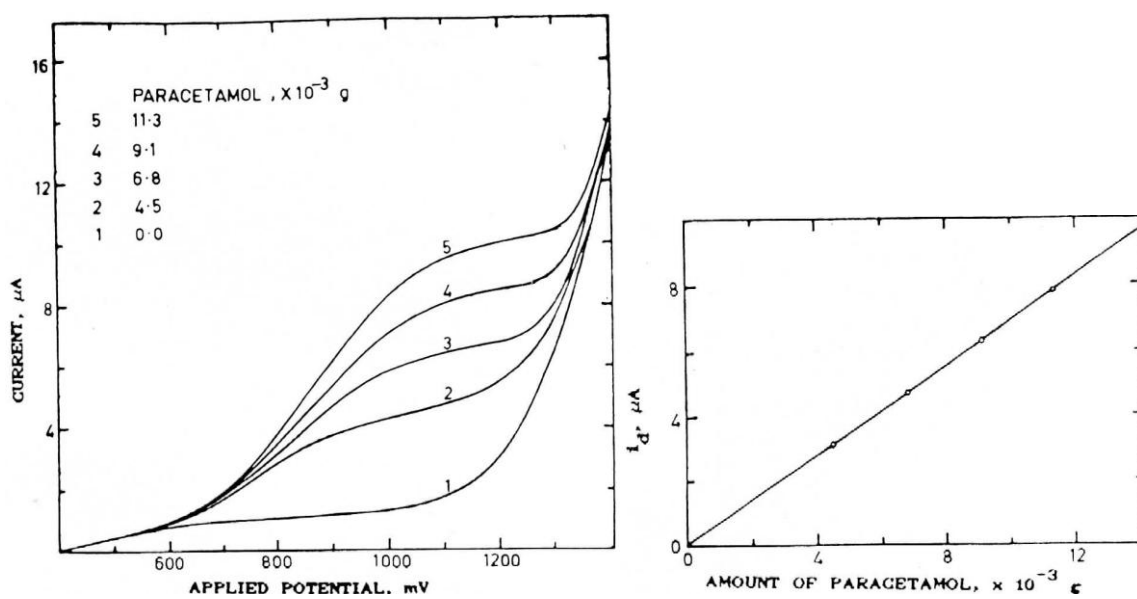
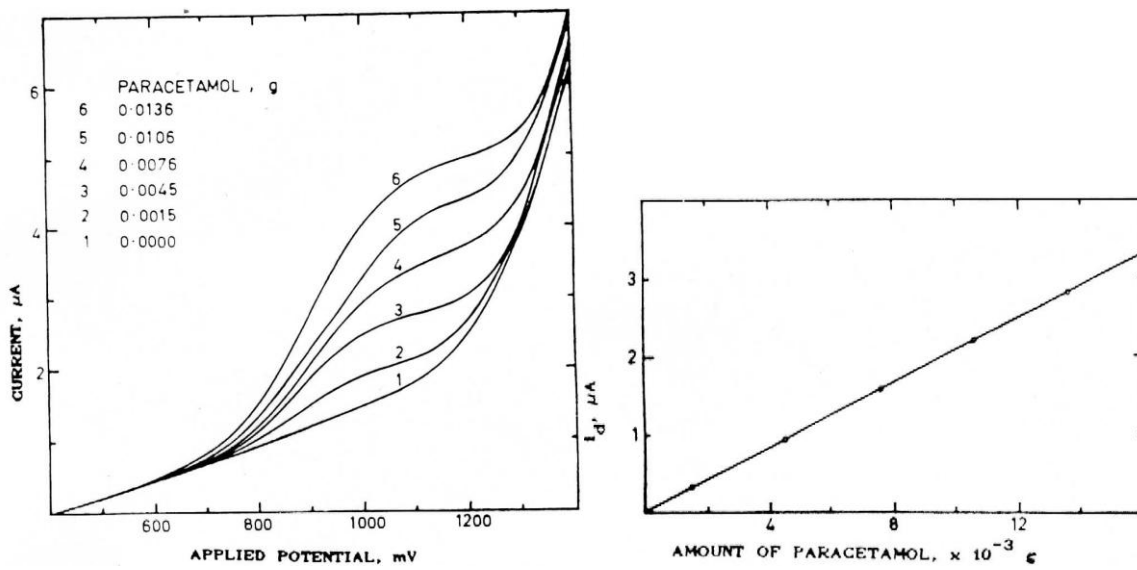


Fig. 5. (a) Calibration polarogram for Paracetamol determination in 1 M CH<sub>3</sub>COOH with 2.5 x 10<sup>-2</sup> % Bromocresol green; (b) Calibration curve for Paracetamol

**Table 2. Polarographic determination of Paracetamol by Calibration method in acetic acid and different Maxima suppressor system**

Medicinal Sample	Weight of Tablet / Capsule material, gm	Weight of Empty Capsule, gm	Amount of Paracetamol per Tablet / Capsule, gm	
			Quoted	Found
<b>In 0.1 M CH<sub>3</sub>COOH &amp; 1 x 10<sup>-6</sup> % Gelatin</b>				
Vikoryl (Alembic) Tablet	0.6195	–	0.5	0.498 ± 0.005
Walagesic (Wallace) Capsule	0.485	0.0946	0.4	0.399 ± 0.007
<b>In 0.1 M CH<sub>3</sub>COOH &amp; 5 x 10<sup>-5</sup> % Fuchsin</b>				
Foracet (Ranbaxy) Tablet	0.6568	–	0.5	0.5 ± 0.1
Fortagesic (Win-Medicare) Tablet	0.662	–	0.5	0.49 ± 0.05
Vikoryl (Alembic) Tablet	0.6195	–	0.5	0.484 ± 0.005
Walagesic (Wallace) Capsule	0.485	0.0946	0.4	0.440 ± 0.008
Malidens (Nicholas) Tablet	0.6952	–	0.5	0.58 ± 0.03
Corbutyl (Roussel) Tablet	1.0389	–	0.65	0.8 ± 0.1
<b>In 0.1 M CH<sub>3</sub>COOH &amp; 1 x 10<sup>-3</sup> % Methyl red</b>				
Corbutyl (Roussel) Tablet	1.0389	–	0.65	0.7 ± 0.1
Fortagesic (Win-Medicare) Tablet	0.662	–	0.5	0.39 ± 0.08
Malidens (Nicholas) Tablet	0.6952	–	0.5	0.50 ± 0.04
Norgesic (Cipla) Tablet	0.4134	–	0.325	0.36 ± 0.07
<b>In 0.1 M CH<sub>3</sub>COOH &amp; 2.5 x 10<sup>-2</sup> % Bromocresol green</b>				
Xeroflam (Helios) Tablet	1.0124	–	0.5	0.5 ± 0.1

Calibration method has been developed and applied for the determination of paracetamol present in some synthetic as well as medicinal samples using acetic acid and different maxima suppressors such as gelatin, fuchsin, methyl red, thymol blue and bromocresol green. The method is strictly empirical, and no assumptions, except correspondence with the conditions of the calibration are made. According to the Ilkovic equation, with all other factors constant.

$$i_d = kC$$

Where k is a constant defined by Ilkovic equation. This relation is the foundation of quantitative polarographic analysis and its general validity is well established. The results of polarographic determination of paracetamol from synthetic and medicinal samples by calibration method are in good agreement with the quoted values. A summary of the results of calibration method is given in table 2. The method is precise as indicated by low values of standard deviations. The

advantages of the application of polarography in the analysis of medicine (paracetamol) are speed, sensitivity, which enables trace analysis to be carried out, and to follow changes in the composition of the preparation, the small sample requirements and selectivity. It is possible to carry out a polarographic analysis even in the presence of colouring matters and comparable amounts of other ingredients such as - Salicylates (aspirin), pentazocine; dextropropoxyphene, codeine and dicyclomie hydrochloride; as in case of Carbutyl, Foracet, Fortagesic, Malidens, Norgesic and Walagesic. Oxyphenbutazone, phenylbutazone and Ibuprofen; in case of Xeroflam. Chlorpheniramine and Phenylephrine; in case of Vikoryl etc.

p-hydroxyacetanilide i.e. paracetamol produces anodic waves at the rotating platinum electrode. The oxidation yields the N-acetyl-p-benzoquinoneimine and represents a irreversible reaction. Polarographically a value of 600-700 mv is found for decomposition potential of

paracetamol, whereas potentiometrically a value of 429 mV is calculated for the same. The presence of oxygen does not affect the wave. The apparent diffusion currents of paracetamol often increase markedly with increasing applied e.m.f. . This is due to the increase of the residual current with increasing applied e.m.f. and when the proper correction is applied for the residual current the corrected diffusion current is found to be practically constant. There are instances, however, in which this correction does not produce a constant limiting current, indicating that the limiting current is not entirely diffusion controlled. Even in such cases, it is found that the limiting current is strictly proportional to concentration when care is taken to measure the current at exactly the same potential with the different concentrations.

**Calibration method using Acetic acid and Gelatin**

Apparent diffusion currents of paracetamol in 0.1 M CH<sub>3</sub>COOH and 1 x 10<sup>-6</sup> % gelatin increases

markedly (without showing proper limiting current) with increasing applied e.m.f. , as in Fig. 1 a. Even in such case, it is found that the diffusion current is strictly proportional to concentration (Fig. 1 b) when care is taken to measure the current at exactly the same potential with the different concentrations. The precision of the linear relation between *i<sub>d</sub>* and C is shown by the data in Table 3. In this case *i<sub>d</sub>*/C is constant to ± 2.8 % over a 4 fold range of paracetamol concentration. Medicinal samples, viz., Vikoryl and Walagesic are found to contain 0.498 ± 0.005 and 0.399 ± 0.007 g paracetamol per tablet which is in good agreement with the quoted values 0.5 and 0.4 g respectively. Both of these tablet seems to contain approved colours, which has no interfering effect. Slight excess values obtained in case of Walagesic may be due to its one of the component Dextropropoxyphene hydrochloride which might be taking part in diffusion phenomenon.

**Table 3. Calibration data for Paracetamol in 0.1 M CH<sub>3</sub>COOH - 1 x 10<sup>-6</sup> % Gelatin and its application to Medicinal Samples.**

Paracetamol, mg		id at 1100 m V		id/C	Amount of Paracetamol per Tablet / Capsule, g	
Taken	Found	Observed	Corrected		Quoted	Found
0	-	0.8	-	-	-	-
0.076	-	2.15	1.35	17.9	-	-
0.152	-	3.525	2.725	18	-	-
0.228	-	4.65	3.85	17	-	-
0.304	-	6.15	5.35	17.7	-	-
				17.6 ± 0.5		
0.28 mg Vikoryl (Alembic) Tablet						
0.226	0.223	4.75	3.95	17.5	0.5	0.494
0.226	0.223	4.75	3.95	17.5	0.5	0.494
0.226	0.225	4.8	4	17.7	0.5	0.499
0.226	0.228	4.85	4.05	17.9	0.5	0.505
				17.7 ± 0.2		0.498 ± 0.005
0.275 mg Walagesic (Wallace) Capsule						
0.226	0.222	4.725	3.925	17.3	0.4	0.392
0.226	0.224	4.775	3.975	17.6	0.4	0.396
0.226	0.228	4.85	4.05	17.9	0.4	0.403
0.226	0.23	4.9	4.1	18.1	0.4	0.406
				17.7 ± 0.3		0.399 ± 0.007

**Table 4. Calibration data for Paracetamol in 0.1 M CH<sub>3</sub>COOH - 5 x 10<sup>-5</sup> % Fuchsin and its application to Medicinal Samples.**

Paracetamol, mg		id at 1100 m V	id/C	Amount of Paracetamol per Tablet / Capsule, g	
Taken	Found			Quoted	Found
0.302	-	1.95	6.4	-	-
0.529	-	3.1	5.9	-	-
0.756	-	4.3	5.7	-	-
0.983	-	5.3	5.4	-	-
1.21	-	6.5	5.4	-	-
			5.8 ± 0.4		
0.993 mg Foracet (Ranbaxy) Tablet					
0.756	0.565	3.25	4	0.5	0.4
0.756	0.61	3.5	5	0.5	0.4
0.756	0.87	4.8	6	0.5	0.6
0.756	0.875	4.825	6	0.5	0.6
			5 ± 1		0.5 ± 0.1
1.005 mg Fortagesic (Win-Medicare) Tablet					
0.759	0.69	3.9	5.1	0.5	0.45
0.759	0.71	4	5.3	0.5	0.47
0.759	0.73	4.1	5.4	0.5	0.48
0.759	0.845	4.675	6.2	0.5	0.56
			5.5 ± 0.5		0.49 ± 0.05

### Calibration using Acetic acid and Fuchsin

In case of 0.1 M CH<sub>3</sub>COOH - 5 x 10<sup>-5</sup> % Fuchsin system the increase in paracetamol concentration results in the linear increase of the wave height (Fig. 2 a). With synthetic as well as medicinal samples good sigmoid C-V curves are obtained (Table 4). Results found are in good agreement with the quoted values with low values of standard deviations.

### Calibration using Acetic acid and Methyl red

Paracetamol can also be determined effectively in 0.1 M CH<sub>3</sub>COOH - 1 x 10<sup>-3</sup> % methyl red system, (Fig. 3 a & b ) method can be applied for its determination in medicinal samples, viz., Carbutyl, Fortagesic, Malidens, Norgesic etc. with good recovery and low values of standard deviation as may be seen from Table 5. Slight excess values obtained in case of Corbutyl may be due to its one of the component Dextropropoxyphene hydrochloride which might be taking part in diffusion phenomenon.

### Calibration using Acetic acid and Thymol blue

While analyzing paracetamol in 2.0 M CH<sub>3</sub>COOH - 1.5 x 10<sup>-3</sup> % Thymol blue solution good calibration polarogram as well calibration graph are obtained (Fig. 4 a & b); but method fails in getting reproducible C-V curve and hence results in medicinal sample such as Corbutyl (Roussel), Norgesic (IDPL) and Seumol Plus (Blue shield) tablets.

### Calibration using Acetic acid and Bromocresol green

In 1.0 M CH<sub>3</sub>COOH - 2.5 x 10<sup>-2</sup> % Bromocresol green medium, it is possible to have well-defined and separated waves relative to the oxidation of paracetamol (Fig. 5 a & b). The method can be applied to the determination of paracetamol in medicinal sample, viz., Xeroflam. With a standard deviation of ± 0.1 (Table 6).

**Table 5. Calibration data for Paracetamol in 0.1 M CH<sub>3</sub>COOH - 1 x 10<sup>-3</sup> % Methyl red and its application to Medicinal Samples.**

Paracetamol, mg		id at 1100 mV	id/C	Amount of Paracetamol per Tablet/Capsule, g	
Taken	Found			Quoted	Found
3	-	2	0.7	-	-
6.8	-	2.95	0.4	-	-
10.6	-	3.9	0.4	-	-
14.4	-	4.8	0.3	-	-
18.1	-	5.75	0.3	-	-
13.365 mg Corbutyl (Roussel) Tablet					
8.362	7.4	3.1	0.37	0.65	0.6
8.362	9.2	3.55	0.43	0.65	0.7
8.362	9.3	3.575	0.43	0.65	0.7
8.362	10.4	3.825	0.46	0.65	0.8
			0.42 ± 0.04		0.7 ± 0.1
18.072 mg Fortagesic (Win-Medicare) tablet					
13.65	8.6	3.4	0.25	0.5	0.32
13.65	9.3	3.575	0.26	0.5	0.34
13.65	10.8	3.95	0.29	0.5	0.4
13.65	13.6	4.625	0.34	0.5	0.5
			0.28 ± 0.04		0.39 ± 0.08
11.561 mg Malidens (Nicholas) Tablet					
8.315	7.6	3.15	0.38	0.5	0.46
8.315	8.1	3.275	0.39	0.5	0.49
8.315	8.4	3.35	0.4	0.5	0.51
8.315	9.2	3.55	0.43	0.5	0.55
			0.04 ± 0.02		0.50 ± 0.04
8.658 mg Norgesic (Cipla) Tablet					
6.807	5.75	2.7	0.4	0.325	0.27
6.807	8	3.25	0.48	0.325	0.38
6.807	8.6	3.4	0.5	0.325	0.41
			0.46 ± 0.05		0.36 ± 0.07

**Table 6. Calibration data for Paracetamol in 1.0 M CH<sub>3</sub>COOH - 2.5 x 10<sup>-2</sup> % Bromocresol green and its application to Medicinal Samples.**

Paracetamol, mg		id at 1100 mV	id/C	Amount of Paracetamol per Tablet / Capsule, g	
Taken	Found			Quoted	Found
4.5	-	3.1	0.689	-	-
6.8	-	4.7	0.691	-	-
9.1	-	6.3	0.692	-	-
11.3	-	7.8	0.69	-	-
			0.691 ± 0.001		
11.468 mg Xeroflam (Helios) Tablet					
5.664	4.85	3.35	0.6	0.5	0.4
5.664	5.1	3.5	0.6	0.5	0.5
5.664	7.1	4.9	0.9	0.5	0.6
			0.7 ± 0.2		0.5 ± 0.1



## CONCLUSION

Paracetamol produces anodic waves at the rotating platinum electrode. The oxidation yields the N-acetyl-p-benzoquinoneimine and represents a irreversible reaction. Polarographically a value of 600-700 mv is found for decomposition potential of paracetamol, whereas potentiometrically a value of 429 mV is calculated for the same. The presence of oxygen does not affect the wave.

Polarographic calibration method has been developed and applied for the determination of paracetamol present in some synthetic as well as medicinal samples using acetic acid and different maxima suppressors such as gelatin, fuchsin, methyl red, thymol blue and bromocresol green. The method is strictly empirical, and no assumptions, except correspondence with the conditions of the calibration are made. Results obtained with synthetic as well as medicinal samples are in good agreement with the quoted values. The method is precise.

It is possible to carry out a polarographic analysis even in the presence of colouring matters and comparable amounts of other ingredients such as - Salicylates (aspirin), pentazocine; dextropropoxyphene, codeine and dicyclomine hydrochloride; as in case of Carbutyl, Foracet, Fortagesic, Malidens, Norgesic and Walagesic. Oxyphenbutazone, phenylbutazone and Ibuprofen; in case of Xeroflam. Chlorpheniramine and Phenylephrine; in case of Vikoryl etc.

### Calibration method using Acetic acid and Gelatin

Apparent diffusion currents of paracetamol in 0.1 M CH<sub>3</sub>COOH and 1 x 10<sup>-6</sup> % gelatin increases markedly (without showing proper limiting current) with increasing applied e.m.f. In this case  $i_d/C$  is constant to  $\pm 2.8$  % over a 4 fold range of paracetamol concentration.

### Calibration method using Acetic acid and Fuchsin

In case of 0.1 M CH<sub>3</sub>COOH – 5 x 10<sup>-5</sup> % Fuchsin system the increase in paracetamol concentration

results in the linear increase of the wave height. With synthetic as well as medicinal samples good sigmoid C-V curves are obtained.

### Calibration method using Acetic acid and Methyl red

Paracetamol can also be determined effectively in 0.1 M CH<sub>3</sub>COOH – 1 x 10<sup>-3</sup> % methyl red system.

### Calibration method using Acetic acid and Thymol blue

While analyzing paracetamol in 2.0 M CH<sub>3</sub>COOH – 1.5 x 10<sup>-3</sup> % Thymol blue solution good calibration polarogram as well calibration graph are obtained; but method fails in getting reproducible C-V curve.

### Calibration method using Acetic acid and Bromocresol green

In 1.0 M CH<sub>3</sub>COOH – 2.5 x 10<sup>-2</sup> % Bromocresol green medium, it is possible to have well-defined and separated waves relative to the oxidation of paracetamol.

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