the complicacy of three-dimensional visualization. The corresponding figure in 2(b) shows the halftone transmittance for the same threshold transmittance. It is to be noted that the patched region has transmittance 1, while the clear area has transmittance 0. For significantly higher transmittance the geometry is always circular.

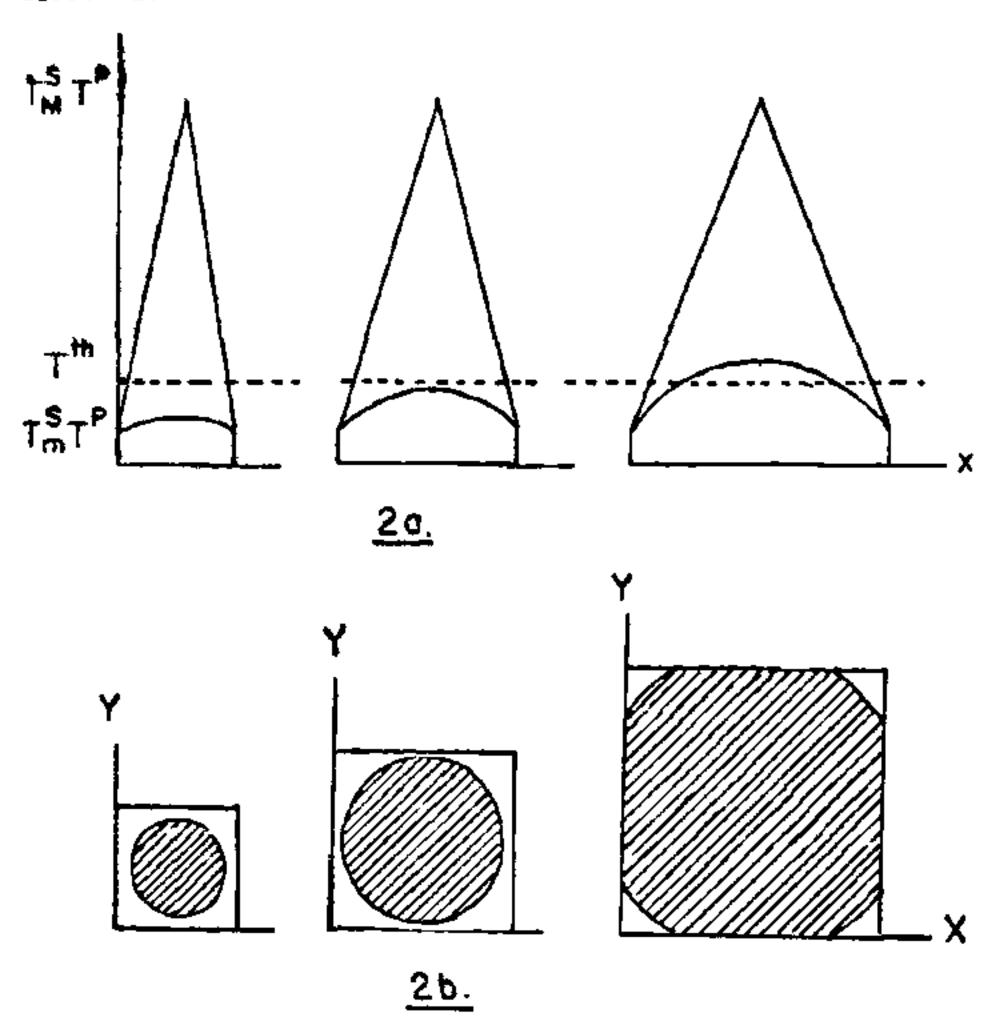


Fig. 2. (a) Transmittances of cell having different cell-periods. (b) Halftone transmittances: patched region has transmittance 1, otherwise 0.

### Conclusion

The dot corruption in circular halftone screen is dependent only on the threshold transmittance. It's

more prominent when the film has a lower threshold value. Dot corruption, for similar reasons, can be expected to occur also in elliptical halftone dots.

For sufficiently sampled images circular shape is preserved to a great extent, and in undersampled image it takes an intermediate geometry between a circle and an octagon. This dissimilarities in the halftone picture will result in correspondingly different output in the spatial frequency plane<sup>10</sup>. Of course a portion of the corruption could be contributed by the scattering phenomenon of the halftone photograph, as described by Yule-Nielsen effect.<sup>11</sup> Circular or elliptical dot corruption could be minimized by selecting higher threshold transmittance and by ensuring a faultless contact printing.

- 1. Wesner, J. W., Appl. Opt., 1974, 13, 1703.
- 2. Kato, H. and Goodman, J. W., *Ibid.*, 1975, 14, 1813.
- 3. Liu, H. K., Goodman, J. W. and Chan, J., *Ibid.*, 1976, 15, 2394.
- 4. and —, Nouv. Rev. Optique, 1976, t. 7, p. 285.
- 5. —, Appl. Opt., 1978, 17, 2181.
- 6. —, Optics Letters, 1978, 3, 244.
- 7. Olof Bryngdahl, J. Opt. Soc. Am., 1978, 68, 416.
- 8. Pappu, S. V., Kumar, C. A. and Mehta, S. D., Curr. Sci., 1978, 47, 1.
- 9. Marquet, M. and Tsujuichi, J., J. Opt. Acta, 1961, 8, 267.
- 10. Smith, R. C. and Marsh, J. S., J. Opt. Soc. Am., 1974, 64, 798.
- 11. Ruckdeschel, F. R. and Hauser, O. G., Appl. Opt., 1978, 17, 3376.

# A SIMPLE COLORIMETRIC METHOD FOR THE DETERMINATION OF PARACETAMOL FROM BIOLOGICAL SPECIMEN

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# ABSTRACT

A simple colorimetric method for the determination of paracetamol is described. The method is based on the measurement of the intensity of crimson colour produced when paracetamol is treated with 10% aqueous sodium hydroxide. No interference is caused by compounds such as phenacetin, aspirin, caffeine, oxyphenbutazone, barbiturates, hydrantoins and dextrapropoxyphen which are present in various analgesic formulations of paracetamol. The method is sensitive to concentrations as low as 25  $\mu$ g/ml paracetamol with a reproducibility of  $\pm 2\%$ .

INCREASING incidences of self-inflicted paracetamol overdosage, single or in combination with other drugs such as oxyphen-butazone, dextropropoxyphen, phenacetin, salicylates and sulphadrugs,

etc., are reported. Paracetamol shares third in rank as a killer drug<sup>2</sup>, barbiturates and aspirin-salicylate being the first and second. The differential absorbance procedure of Routh et al.<sup>3</sup>, because of its relative

specifictiy has been used extensively to determine this drug. However, some drugs such as oxyphenbutazone have been shown to interfere in this estimation4. As a result of oxyphenbutazone, the plasma extract shows the absorption peak at 258 nm and not at 266 nm as expected for the paracetamol. The characteristic "shoulder" on the side of the peak was also absent in presence of oxyphenbutazone. A quick colorimetric method involving nitration of paracetamol to give a yellow colour, further converted to intense orange-red by adding alkali is reported5,6. Although no significant interference was observed in the above colorimetric method for chloropropamide, phenylbutazone, etc., some sulphonamides produced yeollw colour. Sulphathiozole produced greatest interference which decreased in the order sulphadiamidine, sulphasalazine and sulphafurazole<sup>1</sup>. Some other methods reported for paracetamol determination based on blue indophenol dye formation, with various modifications?. Drugs containing aliphiatic, aromatic, nitro, nitrosophenolic -OH or aromatic amino groups interfere in the indiophenol dye methods<sup>8,9</sup>. Paracetamol determination by 2,2-diphenyl-1-pycryl-hydrazyl dye<sup>3</sup>, is found to have its own limitations as the dye reacts with secondary and tertiary aromatic amines and their derivatives. A comparative review of all these methods has been reported by K. Wiener<sup>10</sup>, concluding that at present no single method stands out as offering allround advantages over the other methods.

We have observed that paracetamol on treating with 10% aqueous sodium hydroxide, at elevated temperatures, produces crimson colour having absorption maximum at 463 nm<sup>11</sup>. Beer's-Lambert's law is obeyed for paracetamol concentrations from 25  $\mu$ g/ml to 250  $\mu$ g/ml.

## EXPERIMENTAL

## Reagents

Paracetamol Standard: 0.1% w/v in methanol. Sodium hydroxide: 10% w!v in distilled water.

# Preparation of the Sample

For Blood/urine/plasma/serum/stomach contents: 5.0 ml of the sample is acidified with dilute acetic acid and extracted five times with 50 ml chloroform each time. The chloroform extracts collected together and evaporated to dryness. Dry residue was dissolved in 2 ml methanol added by pipet. This solution is used for the further determination.

# Viscera

50 g minsed viscera is acidified with dilute acetic acid and extracted five times with 100 ml chloroform each time. Chloroform extracts collected together and evaporated to dryness. Residue was dissolved

in 2 ml methanol added by pipet. 1 ml of this solution is further processed as per "General Procedure"

## General Procedure

Take 1 ml of the sample solution in a 10 ml graduated centrifuge tube, add 1 ml 10% NaOH solution and heat in boiling water for 15 minutes. Add 1.5 ml distilled water and allow the mixture to stand for 100 minutes. Make up the volume to 4 ml with distilled water and read on colorimeter at 463 nm "against reagent blank". For "reagent blank" take 1 ml methanol in the test-tube instead of sample and treat under identical conditions.

# Calibration Graph

A series of paracetamol standards were prepared to give concentrations of 100, 200, 300, 400, 500, 600, 700, 800, 900, and 1,000  $\mu$ g/ml. By using the "General procedure" the optical density of the colour produced by each standard concentration was measured. The graph obtained by plotting optical density against concentration of paracetamol gave a straight line passing through origin.

Table I

Recovery experiments done by adding standard paracetamol to cadaveric samples (in vitro)

Biological specimen	Paracetamol added	Amount determined by proposed method	% Recovery
Viscera stomach/ intestine	2·5 mg/100 g	2·243 mg/100 g	97.00
Viscera Liver/ spleen/ kidney	4·0 mg/100 g	3·951 mg/100 g	98.78
Blood	1·2 mg/5 ml	1·192 mg/5 ml	99.30
Urine	1·4 mg/5 ml	1 · 388 mg/5 ml	99 · 14
Stomach Contents	1 · 2 mg/5 ml	1 · 176 mg/5 ml	98-00

#### Discussion

It is observed that phenacetin, acetyl salicylic acid, casseine, oxyphenbutazone, phenylbutazone, barbiturates, hydantoins and dextropropoxyphen which are present in many paracetamol formulations, and therefore, may be present in biological specimen along with

paracetamol, do not interfere in this method. Following compounds which may be of interest to Forensic Toxicologists, also do not interfere in this method: acetanilide, p-nitrophenol, p-nitraniline, p-nitrochlorobenzene, sulphad zine, sulphamethazine and sulphamerazine. p-aminophenol, a metabolite of paracetamol interfere in this reaction which is not extracted during the acidic chloroform extract procedure. The method is found useful for the determination of paracetamol from biological samples. The in vitro recovery of added paracetamol to various biological specimens is found to be between 97 to 99% (Table I).

- 1. Wiener, K., Ann. Clin. Biochem., 1977, 14, 55.
- 2. Registrar-General's Statistics for Death by Poisoning, 1972, Registrar-General for England and Wales, cf: CIBA Foundation Symposium: 26 (New Series), Associated Scientific Publishers, 1974, p. 246.

- 3. Routh, J. I., Shane, N. A., Arredonde, E. G. and Paul, W. D., Clin. Chem., 1968, 14, 882.
- 4. Wiener, K., Longlands, M. G. and Tan, B. H. A., Ann. Clin. Biochem., 1976, 13, 452.
- 5. Glynn, J. P. and Kendal, S. E., Lancet, 1975, 1, 1147.
- 6. Chafetz, I., Daly, R. E., Schriftman, H. and Lomner, J. J., J. Pharm. Sci., 1971, 60, 463.
- 7. Lester, D. and Greenberg, L. A., J. Pharm. and Exptl. Therap., 1947, 90, 68.
- 8. Feigl, F., Organic Spot Test, Elsevier Publishing Company, Amsterdam, The Netherlands, 1956, pp. 154 and 182.
- 9. Davis, D. R., Fogg, A. G., Thornburn Burns, D. and Wragg, J. S., Analyst (London), 1974. 99 (1174), 12.
- 10. Wiener, K., Ann. Clin. Btochem., 1978, 15, 187.
- 11. Sane, R. T. and Kamat, S. S., J. Asso. Offi. Anal. Chem. (In press).

# A NEW TECHNIQUE FOR THE STUDY OF PIEZOELECTRIC PROSPECTING ANOMALIES OVER MODELS,

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#### ABSTRACT

Development of the piezoelectric method as a geophysical prospecting tool needs a practical technique for the evaluation of the piezoelectric activity (PEA) of geological formations. A simple method which makes use of a transient mechanical impulse was utilised in the present study for obtaining a PEA profile across a model quartz vein under laboratory conditions. Besides simplicity, the suggested method offers some practical advantages over existing techniques. The results indicate that a measurable anomaly is obtained over the vein, and that the characteristics of the signal recorded are related to body parameters.

#### INTRODUCTION

THE phenomenon of piezoelectricity of materials, first discovered in the late eighteenth century by the Curie brothers<sup>1,2</sup> presently finds wide application in electronic and related industries. It manifests as electrical polarization in a dielectric when subjected to a mechanical force. Attempts are being made now to adopt this property for geophysical prospecting purposes, viz., for locating vein quartz, other piezomaterials and associated minerals<sup>3,4</sup>.

While the piezoelectric effect is restricted to dielectrics and among them to such crystalline materials as those lacking in a centre of symmetry, many geological specimens were found to display this property4-6.

A constant of proportionality, known as the piezoelectric modulus which relates the electrical polarization intensity with the applied mechanical stress, is generally used to characterize the PEA of substances. For common geological materials, the modulus was reported to vary from  $1.3 \times 10^{-11}$  to  $6.8 \times 10^{-8}$  cgse unit. However, majority of the work on the PEA of geological formations was carried out abroad and data on Indian specimens is practically non existent.

The development of the piezoelectric method as a prospecting tool in Indian conditions needs a priori knowledge of not only the PEA of Indian rocks but also the response of the method in different geological settings.

Laboratory determinations of the PEA modulus and modelling work over PEA bodies as carried out by various workers till now depended upon either the static loading of the sample or continuous excitation of the sample by high frequency vibration<sup>4,7</sup>. Such