

TABLE II.—EFFECT OF ACID USED UPON THE ABSORBANCE MAXIMUM OF THE COLOR DEVELOPED

Sample	N.F. XI Method		Modified Method	
	Absorbance Using HCl	Absorbance Using H <sub>2</sub> SO <sub>4</sub>	Absorbance Using HCl	Absorbance Using H <sub>2</sub> SO <sub>4</sub>
I	0.315	0.324	0.312 (435 m $\mu$ )	0.321 (425 m $\mu$ )
II	0.310	0.320	0.305 (430 m $\mu$ )	0.320 (425 m $\mu$ )
III	0.305	0.315	0.307 (430 m $\mu$ )	0.322 (425 m $\mu$ )

TABLE III.—EFFECT OF CHANGE IN CONCENTRATION OF MAP UPON ABSORBANCE MAXIMUM

MAP in Ca(PAS) <sub>2</sub> , %	Absorbance Maximum Found, m $\mu$	Comment on Curve Characteristics
0.02	425	Sloping plateau occurs
0.05	425	Plateau occurs
0.10	430	Round curve occurs
0.20	430-435	Round curve occurs
0.50	435	Sharp peak occurs
1.00	435	Sharp peak occurs

solution to a second 100-ml. volumetric flask, immersed in an ice bath and containing 50 ml. of water at 0 to 5°, and add 2.5 ml. of sodium nitrite solution (1 in 100). Mix and allow to stand in the ice bath for 3 minutes  $\pm$  5 seconds. Add 25 ml. of sodium carbonate T.S., mix, and place the flask in a water bath at 25° for 15 minutes. Add water to volume, mix, allow the solution to stand at 25° for 3 hours, and filter. Determine the absorbance of the clear solution at the absorbance maximum observed in a range of 425 to 435 m $\mu$  in a 1-cm. cell with a suitable spectrophotometer, using water as the blank. Calculate the per cent of *m*-aminophenol by the formula ( $A - 0.320/1.09$ ), in which  $A$  is the absorbance of

the solution, 0.320 is the absorbance correction factor representing color produced other than by the reaction of *m*-aminophenol initially present, and 1.09 is the factor converting absorbance to per cent of *m*-aminophenol in Calcium Aminosalicylate.

## SUMMARY AND CONCLUSIONS

1. The MAP content found by the proposed modified colorimetric method was comparable to that found by paper chromatography for the Ca(PAS)<sub>2</sub> tested in this study.
2. In the addition of acid to the initial solution in the MAP test, sulfuric acid should be added instead of hydrochloric acid.
3. The color produced by both the N.F. XI and the proposed modified colorimetric method stabilized after standing for 3 hours at 25°.
4. The absorbance spectra obtained for MAP by both the N.F. XI method and the proposed modified colorimetric method showed the absorbance maxima to be between 425 and 435 m $\mu$ .
5. The correction factor for color produced other than by MAP should be 0.320, not 0.372.
6. The N.F. XI method for the determination of MAP in Ca(PAS)<sub>2</sub> is a basically reliable procedure.
7. The value, 1.09, for the conversion of absorbance to per cent MAP was correct.
8. It is recommended that the modifications, as herein described, be adopted by N.F. XI to correct minor sources of error in the determination of MAP in Ca(PAS)<sub>2</sub>.

## REFERENCES

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## Ultraviolet Emission Spectrographic Determination of Trace Palladium to Ascertain the Method Used to Produce Tetracycline

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Ultraviolet emission spectrography may be readily employed to determine trace amounts of palladium in ashed samples of tetracycline. Palladium is not found in samples prepared by direct fermentation and is always found when chlortetracycline is dechlorinated using palladium catalytically.

TWO KNOWN commercial methods are used to produce the antibiotic tetracycline. The catalytic reduction of chlortetracycline using palladium or another of the noble metals (1) and the direct fermentation of the microorganism *Streptomyces aureofaciens* (2).

## METHOD

The general procedure is essentially the carbon arc method of qualitative analysis by Harvey (3) and Nachtrieb (4). The sensitivity of the method was increased by using the slowly rotating slotted disk and stationary emulsion method (5). Using a 35-mg. charge, 0.1 mcg. or 3 p.p.m. can be detected. A charge containing 3.5 mcg. or 100 p.p.m. of palladium produces up to 10 spectral lines of the metal in the region 3500 to 2450 Å.

The 600–800° ash cake is ground fine in the porcelain crucible. The exposure is 3 min. as required by the 11-slot disk (5). The spectral line

palladium 3242.703 Å. is used for detecting the least amount of palladium, even though it is theoretically not the strongest. It is conveniently close to copper 3247.540 Å. for positive identification and has very little interference from a carbon line.

The length, width, and density of the line are mentally integrated into an order of magnitude value (3, 4) and applied to the least detectable concentration of 3 p.p.m. The least detectable concentration is different in each ashed sample. It depends greatly upon the amount of reduction in weight by dry ashing at 600 to 800°.

## REFERENCES

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Received June 3, 1961, from American Cyanamid Co., Central Research Division, Stamford, Conn.

Accepted for publication February 26, 1962.