sampling errors and permitted direct comparison of the methods. The data for the acid pretreated oils are the averages of fully concordant results obtained by use of both reactions.

The values obtained by use of the aniline reaction emphasize the fact that low results are obtained at 440 mμ for most hydraulic-, screw-pressed, and solvent-extracted oils. Other oils, such as prepressed and direct solvent-extracted, yield comparable results at either 440 or 450 mμ. In practically all cases gossypol estimates with the photoelectric colorimeter (470 filter) tended to be high.

For the p-anisidine method, consistent results were obtained by use of either a spectrophotometer at 460 mμ or a colorimeter (470 filter). In agreement with previous data (Table II) analysis of crude oils by use of the aniline reaction and reading the absorption at 450 mμ or the p-anisidine reaction at 460 mμ gave comparable results which are in good agreement with those for either reaction applied to oxalic acid pretreated crude oils.

From consideration of the data it would appear that use of the p-anisidine reaction at 460 mμ or the aniline reaction at 450 mμ is equally suitable for the analysis of gossypol pigments in crude cottonseed oils. However a serious objection to the use of the aniline reaction is the requirement that measurements be made at 450 mμ, rather than at the dianilinogossypol maximum at 440 mμ, in order to avoid over-correction for background absorption. This qualification will lead to a lowering of the reproducibility of the method, particularly when photoelectric colorimeters are used. Since p-anisidine-gossypol exhibits a second absorption maximum at 467 mμ, where modified gossypol pigments do not interfere with the background correction, the p-anisidine procedure is preferable from the standpoint of accuracy and precision. With a pretreatment of crude oils with oxalic acid in methyl ethyl ketone prior to analysis, measurements can be made at the principal absorption maximum of 440 mμ in the case of aniline-gossypol and at 447 mμ for p-anisidine-gossypol. However the added complexity of analysis does not seem to justify the use of the oxalic acid pretreatment.

Summary

The p-anisidine method for the determination of gossypol in crude cottonseed oils has been reinvestigated and modified to make it applicable to all crude oils obtained by the newer methods of processing cottonseed. The modifications included a change in the composition of the solvent, a higher reaction temperature, and the use of a more concentrated p-anisidine reagent. The modified method was found satisfactory where different colorimeters and spectrophotometers were used for measuring the color developed.

Comparison of aniline and p-anisidine as reagents for the analysis of gossypol pigments showed that the presence of modified gossypol in some crude oils resulted in an over-correction for background absorption and led to significant errors when aniline was used as the color development agent.

REFERENCES

1. American Oil Chemists' Society, "Official and Tentative Methods of Analysis," Ed. 2, rev. to 1955. Chicago, 1946-1955, Tentative Methods Ba 7-55 and Ba 8-55.
2. Dechary, J. M., Kupperman, R. P., Thurber, F. H., and O'Connor, R. T., J. Am. Oil Chemists' Soc., *31*, 420-424 (1954).
3. King, W. H., and Thurber, F. H., J. Am. Oil Chemists' Soc., *30*, 70-74 (1953).
4. Miller, W. J., J. Am. Oil Chemists' Soc., *32*, 29-33 (1955).
5. Pons, W. A. Jr., Thurber, F. H., and Hoffpauir, C. L., J. Am. Oil Chemists' Soc., *32*, 98-103 (1955).
6. Pons, W. A. Jr., Murray, M. D., LeBlanc, M. F. H. Jr., and Castillon, L. E., J. Am. Oil Chemists' Soc., *30*, 128-132 (1953).
7. Pons, W. A. Jr., Hoffpauir, C. L., and O'Connor, R. T., J. Am. Oil Chemists' Soc., *28*, 8-12 (1951).
8. Pons, W. A. Jr., Hoffpauir, C. L., and O'Connor, R. T., J. Am. Oil Chemists' Soc., *27*, 390-393 (1950).
9. Pons, W. A. Jr., Murray, M. D., O'Connor, R. T., and Guthrie, J. D., J. Am. Oil Chemists' Soc., *25*, 308-313 (1948).
10. Pons, W. A. Jr., and Hoffpauir, C. L., J. Am. Oil Chemists' Soc., *32*, 295-300 (1955).
11. Thurber, F. H., Vix, H. L. E., Pons, W. A. Jr., Crovetto, A. J., and Knoepfler, N. B., J. Am. Oil Chemists' Soc., *31*, 384-388 (1954).
12. Williams, P. A., Hadden, R. P., Hall, C. M., Castillon, L. E., Guice, W. A., O'Connor, R. T., and Boatner, C. H., J. Am. Oil Chemists' Soc., *26*, 28-34 (1949).

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Letter to the Editor

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Lancaster, Bitner, and Beal in a recent paper (1) have described a procedure for determining the induction period in an atmosphere of oxygen as a measure of the stability of an oil. They comment on the small amount of attention required and, by implication, suggest that such a method is superior to the so-called Active Oxygen Method.

The principle of this procedure was suggested by us in 1942 (2), and apparatus of this type has been in constant use in these laboratories for 16 years. Our determinations are made at the temperature of boiling water in an atmosphere of oxygen, and the oxygen absorption is indicated by a continuous record of the mercury level in a manometer. The apparatus is in current use in several other laboratories in Great Britain.

As a result of our experience we have no hesitation in agreeing with the conclusions of Lancaster and his colleagues. The apparatus is eminently satisfactory for routine work, the services of an analyst are not required, and the results are obtained automatically without attention, whatever the length of the induction period may be.

REFERENCES

1. Lancaster, E. B., Bitner, E. D., and Beal, R. E., J. Am. Oil Chemists' Soc., *33*, 36 (1956).
2. Sylvester, N. D., Lampitt, L. H., and Ainsworth, A. N., J. Soc. Chem. Ind., *61*, 165 (1942).

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