

(7). The non-lipid extraction is in the reverse order being the greatest with the 90% ethanol perhaps in part because of the lower water solubility of both protein and carbohydrates in higher ethanol concentrations. The former may result from the denaturing effect of the ethanol such as was shown in previous studies in this laboratory on ethanol-extracted cottonseed (3).

The quality of the oil is shown in Table II. Except for the slightly higher phospholipid, and correspondingly lower neutral oil, content in the oils extracted by the 90 and 95% ethanol, the oils show no significant differences. The protein content of the different meals (Table II) is not significantly different but far below the over 70% value given by Mustakas et al. (8) for ethanol-washed hexane-extracted meal. Apparently when used as a solvent the ethanol preferentially dissolves the oil and cannot take up as much carbohydrate material.

Conclusions

In the extraction of soybean oil by aqueous ethanol lipids are removed more rapidly and non-lipids less rapidly as the concentration of the ethanol increases.

• Letter to the Editor

Tetracyanoethylene Adduct of *Trans,Trans*-9,11-Octadecadienoic Acid

SEVERAL Diels-Alder adducts of *trans,trans*-9,11-octadecadienoic have been reported (1) from this laboratory. This acid reacts readily with nitroethylene, β -nitrostyrene, acrylic acid, acrylonitrile, acrolein, methacrolein, methyl vinyl ketone, methyl vinyl sulfone, and propargylic acid. We have now prepared the adduct with tetracyanoethylene (TCNE). This dienophile reacts readily with a wide variety of dienes (2). Reaction with *trans,trans*-9,11-octadecadienoic acid proved to be equally facile. The course of the addition could be followed by typical color changes (2) from the initial green of the tetrahydrofuran (THF) solution of TCNE through the dark red of the intermediate pi-complex to the final light yellow solution of the adduct. After recrystallization, the adduct was a slightly off-white crystalline solid, melting at 69.5–71°C. The infrared spectrum was consistent with the Diels-Alder structure; absorption bands showed the presence of a carbocyclic ring, *cis*-carbon-carbon unsaturation, and nitrile and carboxylic acid groups.

Experimental

Trans,trans-9,11-octadecadienoic acid (4.2 g, 0.015 mole) and TCNE (1.9 g, 0.0148 mole) were each dissolved in 7.5 ml of THF. The acid was only partially soluble in this amount of solvent. The TCNE solu-

tion was dark green. After the two solutions were mixed, the mixture turned dark red and slowly became a light yellow (in about 5 min). The THF was evaporated under vacuum to leave a viscous gray-green residue. Ten milliliters of pentane-hexane was added and the mixture stirred until the adduct solidified to a white powder, which was filtered to give 5.8 g of solid. Recrystallization from benzene-pentane-hexane gave slightly off-white mica-like crystals, mp 69.5–71°C.

Anal Calcd. for $C_{24}H_{32}N_4O_2$: C, 70.55; H, 7.90; N, 13.72. Found: C, 70.43; H, 7.93; N, 13.60.

Acknowledgment

The authors are indebted to Dr. T. L. Cairns for the TCNE used in this work, to D. Kuehn for microanalyses, and to G. E. McManis for spectral analyses.

W. R. MILLER AND J. C. COWAN
Northern Regional Research Laboratory
Peoria, Illinois

REFERENCES

- Teeter, H. M., J. L. O'Donnell, Wilma J. Schneider, L. E. Gast, and M. J. Danzig, *J. Org. Chem.*, **22**, 512–514 (1957).
- Middleton, W. J., R. E. Heckert, E. L. Little, and C. G. Krespan, *J. Am. Chem. Soc.*, **80**, 2783–2788 (1958).

[Received May 14, 1962]

[Received November 3, 1961]