velopment was safe from volatility losses and saved reaction time. In all probability, higher chloramine-T concentrations, coupled with appropriate adjustments in the amount of arsenite used, would allow complete chlorination in a much shorter time, perhaps as little as 30 minutes.

Sodium arsenite had to be introduced to reduce a significant reagent blank apparently caused by excess chloramine-T. [Sodium arsenite has been used previously (*12*) as a titrant to determine chloramine-T, using starch–potassium iodide paper as an external indicator.] Reduction in background had to be balanced against a reduction in developable color, since it was found that a twofold excess of arsenite started to cause reductions in developed colors. Color reduction was probably caused by attack on the intermediate cyanogen chloride already formed. With longer times allowed for reaction of chloramine-T with samples, the reagent blank diminished, but was still worth correcting for. As demonstrated by Epstein (*5*), ferric chloride acts as a catalyst in the conversion of thiocyanate to cyanogen chloride. In early experiments with corn samples to which thiocyanate had been added, those chlorinated in the presence of ferric chloride gave two and a half times the color density of those treated without this catalyst. Large quantities had no more effect than the indicated amount of ferric chloride.

Sodium acid succinate buffer used to alter the sample from a very slight acid condition (pH 6.5 to 6.8) to pH 4.7 was successful in giving an absorbance increase of 22% for thiocyanate standards. A potassium acid phthalate buffer of very close pH (4.2) produced very little enhancement.

At first, microdiffusion was permitted to proceed with agitation once every few minutes and this appeared to give maximum transfer in 30 minutes. Later work showed that continuous, gentle rocking doubled the slope of the calibration curve for the same 30-minute transfer period. Because of the beginning of a slow decomposition of the developed color after about 40 minutes, it was not possible to determine how long complete transfer, without agitation, would take. A value extrapolated from "stagnant" microdiffusion experiments would be between 70 and 110 minutes for complete transfer.

While chloramine-T and bis(pyrazolone) solutions can be successfully stored in a very cold refrigerator (30° F.) for several days, the over-all sensitivity will suffer somewhat. The bis(pyrazolone) preserves the colored reaction product of thiocyanate, pyridine, and pyrazolone; however, the unreacted mixed pyrazolones reagent deteriorates quickly and should be mixed within 0.5 hour of use. Refrigeration does not seem to help and reagent blanks are needed to correct for the build-up of background color even when used as suggested. After microdiffusion is completed the color is stable for at least 15 minutes and slow detectable fading is evident only after 25 minutes. In order to minimize the above variables, two standards and two reagent blanks were run with each batch of five or six samples.

The sensitivity of the method is readily increased by an order of magnitude by extracting the developed color into *n*-butyl alcohol (*1*), but this seemed unnecessary because of the large quantities of naturally occurring thiocyanate found in corn.

Evidence that the substance being determined is actually thiocyanate is available from a comparison of spectra (Figure 1) of the colored reaction products produced by pure thiocyanate in water and by untreated corn. The original use (*5*, *8*) of the pyridine-pyrazolone system was for color development directly in the same solution in which the thiocyanate had been treated with chloramine-T. The spectrum of such a reaction is also shown for comparison. The small deviations in the shape of these curves as well as the minor shift in peak location are unexplained.

The analytical peak had been reported at both 620 (*8*) and 630 mμ (*5*) by different workers.

Corn Grown on Amitrol-T Treated Plots. Experimental plots were treated with 4 pounds per acre of Amitrol-T, 1 month before planting and plowed 3 days before planting. Corn from these plots as well as from duplicate, untreated plots was harvested about 15 weeks after planting. Analyses of these samples (Table I) showed no detectable increase of thiocyanate over the naturally occurring levels. Use of the 2-pound rate should provide an even smaller opportunity for residue pickup.

A sample of fresh frozen corn purchased at a local food market showed a thiocyanate level in the same 60 to 80 p.p.m. range as did the test samples.

The natural occurrence of thiocyanate in plants has been recorded before as has been the occurrence of thiocyanate in normal human beings: blood (1.6 to 15 p.p.m.) (*10*), urine (8 to 20 p.p.m.) (*3*), gastric juice (11 to 40 p.p.m.) (*3*), spinal fluid (0.06 to 0.2 p.p.m.) (*3*), saliva (mean value 117 p.p.m.) (*6*), and breast milk (4 to 5 p.p.m.) (*14*). Over 100 samples of milk from Jersey cows showed 4 to 5 p.p.m. (*14*). Gemeinhardt (*7*) reported 0.03 to 9.5 p.p.m. of thiocyanate in 55 different plants, mostly common fruits, vegetables, and grains. His values are reported on an as received basis and make no allowance for moisture content. It would appear from the above that small quantities of thiocyanate are a normal component of our diet and of our bodies.

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Correction

Determination of Heptachlor Epoxide in Fat and Milk

In this article by Charles F. Meyer, Marshall A. Malina, and Percy B. Polen [J. Agr. Food Chem. **8**, 183 (1960)], under the Reagents section, the 25th line of column two on page 183 should read "Add an equal volume of butyl Cellosolve." The word Cellulose was incorrectly used in place of Cellosolve.