

Bricker C E & Johnson H R. Spectrophotometric method for determining formaldehyde. *Ind. Eng. Chem.* 17:400-2 1945.
[Research Laboratories, Heyden Chemical Corporation, Garfield, NJ]

A selective spectrophotometric determination of microgram quantities of formaldehyde is described. The sample is treated with chromotropic acid and after adding sulfuric acid and heating in boiling water, the reaction mixture is diluted. The intensity of the purple color is proportional to the quantity of formaldehyde present. [The *SCI*® indicates that this paper has been cited over 180 times since 1961.]

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"In the Fall of 1943, I accepted a position with the Heyden Chemical Corporation and was assigned the problem of finding a method that would be rapid, reliable, and reproducible for the determination of trace amounts of formaldehyde in pentaerythritol (PE). PE was being used by the armed services to make pentaerythritol tetranitrate (PETN), one of the explosives used in blockbusters during World War II. Since PE is prepared from acetaldehyde and excess formaldehyde, it was suspected that a trace impurity of formaldehyde in the PE would contribute to the instability of the PETN. Any formaldehyde present in the PE was not present as free formaldehyde but had formed a compound with the parent compound.

"The more familiar methods for determining formaldehyde, Schiff's Reagent for the colorimetric determination and the precipitant dimedone for the gravimetric determination, had been investigated extensively and were found to lack sensitivity and/or reproducibility. These

methods also required the separation and removal of the

formaldehyde from the PE before these tests could be applied. One reference in Feigl's book caught my attention, (p. 327)¹ This spot test indicated that formaldehyde plus chromotropic acid in the presence of sulfuric acid produced a purple color.

"As soon as some chromotropic acid (1,8-dihydroxynaphthalene, 3,6-disulfonic acid) was obtained, Hilding Johnson, my associate in the Analytical Research Laboratory, and I studied the experimental parameters that affected the development of the purple color between formaldehyde and chromotropic acid. In a short time, we optimized the variables and with these conditions, no other organic compound was found to give a purple color with chromotropic acid and only a few compounds prevented the formation of the color with formaldehyde. Since the color development was carried out in strong acid, the formaldehyde that was combined with the PE was released and thereby eliminated the necessity for a separations step.

"I submitted a paper based on this method to be presented at the New York meeting of the American Chemical Society in September, 1944. At the time of the presentation, a hurricane was at its full fury, and the top floor of the Hotel Taft, where the analytical section was held, was swaying noticeably. Since this was my first presentation at an American Chemical Society meeting, I attributed the shaking of my knees to the weather!

"This method for the detection of formaldehyde not only solved the immediate problem of the analysis of pentaerythritol for trace quantities of formaldehyde but has subsequently provided the basis for the determination of trace quantities of any organic compound that will produce formaldehyde "

1. Feigl F. *Spot tests*. New York: Elsevier, 1939. 462 p.