

Menn J J, Erwin W R & Gordon H T. Color reaction of 2,6-dibromo-N-chloro-p-quinoneimine with thiophosphate insecticides on paper chromatograms. *J. Agr. Food Chem.* 5:601-2, 1957.

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A simple and rapid detection paper chromatographic method for sulfur-containing phosphate ester insecticides was found by spraying the developed chromatogram with 2,6-dibromo-N-chloro-p-quinoneimine. As low as 1 µg of material was detected for some of the insecticides studied. This color reaction is useful for rapid identification for a wide variety of thiophosphate insecticides from plant or animal residues. [The SC<sup>1</sup>® indicates that this paper has been cited in over 115 publications.]

## A Chromogenic Reagent for Thiono and Thioether Compounds

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In 1954 I enrolled at the University of California, Berkeley, as a PhD candidate in insect toxicology under the guidance of Professor William M. Hoskins and in an informal professional association with Dr. Harold T. Gordon, biochemist, and Mr. Wallace Erwin, chemist, in the Insect Toxicology Laboratory. Those were the heydays of organophosphorus ester (OP) insecticides, which dominated the field of chemical insect control for two decades, until the late 1970s. In the 1950s we were still in the infancy period of metabolism studies, paper chromatography, and detection methodology.

I was engaged in research on metabolism of OP insecticides in insects. In the course of this work, I was searching for a chromogenic re-

agent for OP insecticides containing thiosulfur moieties. At the same time, Dr. Gordon was developing spot reactions for sulfur-containing amino acids on paper chromatograms with the Gibbs reagent 2,6-dibromo-N-chloro-p-quinoneimine (DCQ) based on the reducing properties of the Gibbs reagent in aqueous systems. This reagent in our hands proved to be remarkably successful in nonaqueous systems in detecting thionosulfur and thioether sulfur moieties on paper chromatograms in submicrogram quantities by employing simple and rapid detection procedures involving spraying chromatograms with a DCQ solution followed by mild heating.

This procedure proved to be widely applicable to the whole class of OP pesticides. Furthermore, it proved highly useful in detecting parent and *in vivo* formed oxidative metabolites of these insecticides in insects<sup>1</sup> and plants.<sup>2</sup>

DCQ gained wide usage in pesticide detection studies as a quick qualitative and/or semi-quantitative detection method in plant, animal, and soil residues. The discovery of DCQ was also associated with early developments in reverse-phase paper chromatography in our laboratory.<sup>3</sup> These same systems are still widely used in reverse-phase high-performance liquid chromatography (HPLC). The versatility of DCQ was applied also to non-OP compounds; it was helpful in elucidating a new metabolic pathway for thiophenol in the rat.<sup>4</sup>

Although advanced analytical methods in residue analysis, including gas chromatography radiotracer and HPLC, have largely superseded the use of chromogenic agents such as DCQ, the latter is still a highly useful analytical aid in synthetic studies with OP insecticides<sup>5</sup> and metabolic studies.<sup>6</sup> Undoubtedly, DCQ will continue to find utility as an analytical aid even now, 32 years since its discovery.

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