

## Thin-layer chromatography of 2,4-dinitrophenylosazone homologs of vicinal dicarbonyls

Recent column partition<sup>1,2</sup> and adsorption<sup>3</sup> methods have facilitated the partial resolution of 2,4-dinitrophenylosazone (DNPO) mixtures. These methods have also been supplemented by a thin-layer chromatographic (TLC) technique that will separate the osazones of vicinal dicarbonyls into classes<sup>4</sup>. The purpose of this communication is to describe a TLC method that effects separation of a series of DNPO

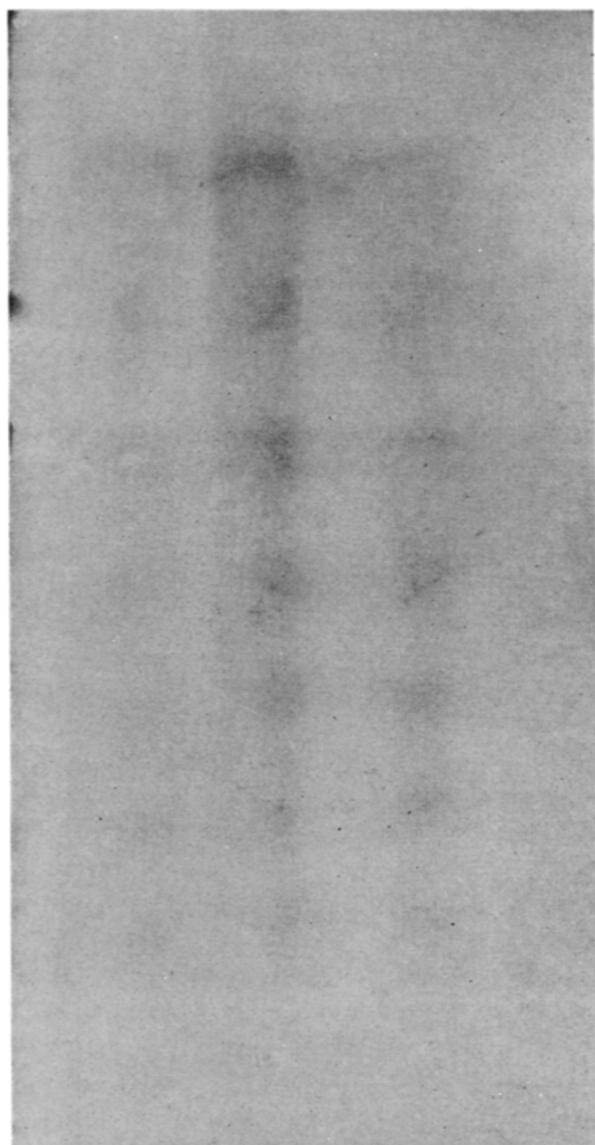


Fig. 1. Thin-layer chromatoplate of 2,4-DNPO of  $C_4$ - $C_{10}$   $\alpha$ -ketoalkanals. Solvent front: 15 cm. Development time: 8 h.

homologs. The technique is a slight modification of a reversed-phase TLC system<sup>5</sup> for the separation of homologous series of 2,4-dinitrophenylhydrazones of monocarbonyl compounds.

### Experimental

Thin-layer plates coated with silica gel G in a 250  $\mu$  layer were dried in an oven at 100° for 1 h, cooled, then dipped into a solution of 10% Shell Ondina 27\* mineral oil in petroleum ether. Upon evaporation of the petroleum ether the plates were spotted with the DNPO mixture approximately 1/4 in. from the base of the plate in the usual manner. The plates were developed for about 7 h (or until sufficient separation had been accomplished) in a system of dioxane-water (6:4). During development, one end of the plate was exposed to the atmosphere in the manner described by LIBBEY AND DAY<sup>5</sup>. This technique negates multiple development.

Fig. 1 illustrates the resolution of an homologous series of  $\alpha$ -ketoalkanal. Since the system is reversed-phase chromatography, the higher the number of carbon atoms in the parent chain the slower the migration rate. 2,3-Diketones were also resolved by the technique. In our laboratory, a mixture of the C<sub>4</sub>, C<sub>5</sub>, C<sub>7</sub> and C<sub>8</sub> diketone derivatives, developed for 7 h, showed resolution approximating that of the  $\alpha$ -ketoalkanal.

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\* *Note added in proof.* Since submitting this note it has come to the authors' attention that Shell Oil Company no longer manufactures mineral oils. Nujol, a product of Plough, Inc., New York, has been evaluated as a substitute stationary phase and found to perform adequately. It is assumed that comparable grades of mineral oil also may be employed.