SPECIAL CHEMICAL ANALYSIS

Jacob Sandoval

DEVELOPMENT DIVISION

OCTOBER - DECEMBER 1971

*Normal Process Development
Endeavor No. 223*

MASTER

Mason & Hanger-Silas Mason Co. Inc.
Pantex Plant

P. O. BOX 647
AMARILLO, TEXAS 79105
806-335-1581

operated for the
ATOMIC ENERGY COMMISSION
under

U. S. GOVERNMENT Contract DA-11-173-AMC-487 (A)

NOTICE

~~This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately-owned rights.~~

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

SPECIAL CHEMICAL ANALYSIS

Jacob Sandoval

DEVELOPMENT DIVISION

Thin-layer chromatography, TLC, a discriminating qualitative analytical tool, is indispensable to the researcher in explosives, e.g., in establishing product composition and purity.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

October - December 1971
Endeavor No. 223
Acct. No. 22-2-44-02-223

Section Z

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

8

SPECIAL CHEMICAL ANALYSIS

ABSTRACT

Thin-layer chromatography, TLC, is an indispensable tool to those involved with high explosives, e.g., the research chemist. Through TLC evaluation he is able to establish the purity of the products he is synthesizing or of the reagents involved in the synthesis.

DISCUSSION

Thin-layer chromatography can be utilized in making qualitative and quantitative determinations of organic as well as inorganic matter. The utility of this analytical technique is extensive, suggesting that an abbreviated description can be useful here.

Thin-layer chromatography, like all chromatographic separating methods, involves a mobile phase passing over a stationary phase. The mobile phase transports different materials, at different speeds, in the direction of the flow. Each migrating substance travels independently of any other substance present.

During the migration, individual material particles oscillate between the two phases constituting the chromatographic system. After each entry and subsequent withdrawal from the mobile phase, substances reside there for a certain period of time. The sum of these time intervals is termed the "residence times." Substances migrate through an adsorbing layer simultaneously only if their residence times are identical.

Substances can be transported only while in the mobile phase; thus, the difference in propagation of substances results from the difference in "residence times of the corresponding particles."

The choice of adsorption or transportation media may be dictated by the characteristics of the compounds to be separated. Thus the properties of the compound under investigation must be known in order to select the proper solid and liquid phases. If the characteristics of the compound are unknown, they must be established. This involves additional work and often requires detailed study.

Solutions of the compounds to be separated, as well as of the known standards, are applied near the base of the carrier plate. The plate is then lowered into the development tank which contains just sufficient solvent to provide a saturated atmosphere. The solvent front ascends uniformly throughout the plate.

After the plate is developed it is sprayed with an appropriate agent. The spray stains the resulting separated compounds, whose concentrations appear as spots on the adsorbent. Comparison of the migration distance of the spots—unknown versus standards—is used in identification of compounds as well as in establishing product purity.

The spots appearing on the plate are indicative of the purity of the product. A single spot indicates no adulterant. Impurities, if present, appear above or below the main spot, depending on their residence times.

Occasionally, any one of these impurities may be of interest to the researcher. The product of interest can be obtained for characterization by scraping the material off one or more plates and extracting it from the scrapings with a suitable solvent.

TLC chromatograms are sensitive to mechanical shock. Furthermore, the spots tend to fade with time. For these reasons, and because the carrier-plate glass can be used repeatedly, photographs of the chromatogram are made and the pictures saved for documentation. Fig. 1 is a record of a chromatogram involving the separation and analysis of TATNB, BTF, FEFO, TNT, PETN, and DiPEHN.

In this laboratory TLC is used exclusively in qualitative evaluation of compounds; however, it may also be employed in making quantitative determinations of chemical compositions. In quantitative analysis numerous variables exist which need to be standardized and maintained constant.

- a. *Particle size*
- b. *Plate thickness*
- c. *Spot size or color density*
- d. *Charge mass, etc.*

The investigator has not used TLC in quantitative analysis because of the tedium, the present uncertainty of results, and because faster and more reliable quantitative analytical procedures are usually available.

TLC as an analytical tool became appreciated as the capabilities became evident. TLC techniques for the separation and identification of numerous explosives were established and written. Although some of these procedures have since been slightly modified, they continue to be used in qualitative analysis of RDX, HMX, TNT, Comp B, Cyclotol, PETN and its homologs (along with esters of the same), CATCP, CPCP, CPZP, etc.

As new explosives are developed additional TLC techniques come into existence. During the past quarter year the following compounds have been separated and analyzed: FEFO, AFNOL, TATNB, BTF, DNPA, and RX-08.

TLC is not being pursued as an academic effort; instead, this analytical capability is being utilized as a service to the research chemist, the chemical engineer, and the process engineer.

FUTURE WORK; COMMENTS; CONCLUSIONS

"Special Chemical Analyses" are not done on a pre-established time schedule. Samples are evaluated on a need basis; thus, the activity is contingent on the number and the nature of samples submitted for analysis.

As new explosives are synthesized, procedures for their analysis are modified or developed.

TLC is available to the research chemist and to the Manufacturing and Quality Divisions' personnel and it is in frequent use for many explosives and intermediate products.

1. TC1TNB	4. FEFO
2. TATNB	5. TNT
3. BTF	6. PETN
	7. DiPEHN

- A. Plate sprayed with ammonium sulfide. Picture taken while plate wet
 B. Picture of plate A after spray dried
 C. Plate resprayed with Greiss reagent

Fig. 1. TLC Plate: Spray Effect on Spot Development