TECHNICAL INFORMATION

Master Questioned Document Kit

Catalog No. MQDA500

INTRODUCTION

The MQDA500 is one of the most complete kits available for the examination and analysis of questioned documents. The kit includes equipment for color analysis of inks, restoration of altered writing, indented writing, indented writing restoration and thin layer chromatography identification of ballpoint and felt tip pen ink. A combination longwave and shortwave UV light, 5X magnifier, optical analysis instrument and questioned document analysis plates are also included.

PRECAUTIONS

- Consult appropriate Material Safety Data Sheets (MSDS) found on our website at www.sirchie.com/support.
- Corrosive/Oxidizer. Harmful if swallowed or inhaled. Skin and eye irritant. If contact made with skin, wash with soap and water; if contact made with eyes, flush with plenty of water for approximately 15 minutes—seek medical attention.

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- Extremely Flammable! Do not use Mobile Solvents (A, B, C), No. ERFT1, or No. EINK2 around heat, open flames or sparks—dispose of empty containers according to Local, State, and Federal regulations.

BACKGROUND

Thin layer chromatography is the technique of separation and identification of chemical composition by a moving solvent on thin layers of silica gel, alumina or other absorbent materials. A drop or spot of solution containing the substance to be separated is placed near one end of a thin layer sheet. The solvent is allowed to evaporate, leaving a spot of the substance. The sheet is then immersed upright in a suitable solvent, the spotted end down with the spot just above the solvent. The solvent will flow up the sheet because of the capillary action. It is allowed to flow almost to the top of the sheet. The solvent flows over and past the spot of substances to be separated. As it flows, it dissolves and carries along the substances from the spot, each substance moving at a different rate from the others. After the solvent has flowed the prescribed distance, the plate is dried. Location of the spot is accomplished by ultraviolet scanning or spraying with a suitable reagent.

MQDA500 Lid Contents

Principle of Separation

There are two forces in play as the solvent moves over the coating of the sheet and the spot:

Tray 1 Contents

Tray 2 Contents

Tray 3 Contents

Propelling Force and Retarding Force. The Propelling Force acts to move the substances and displace them in the direction of the flow of solvent. Two factors concerned in this force are the solvent flow and the solubility of the substance in the solvent.

When the solvent flows, it carries all dissolved substances with it. If the substance is very soluble in the moving solvent, and if no retarding forces are present, the substance will be found at the solvent front (furthest point reached by the solvent). On the other hand, if the substance is very insoluble, it will remain at the point where it was applied to the sheet. If the solvent dissolved all substances in the applied spot completely, the would all move to the solvent front. This would get us nowhere if it were not for the effect of solubility and retarding forces at play.

Few substances have the same solubility in any one solvent. The solubility of a substance in the flowing solvent is a force that tends to displace it from the gel on the sheet. The more soluble the substance, the faster it will move. More soluble substances will move faster than the less soluble ones. Choice of solvent for a separation on a thin layer of chromatogram is an important factor. These, along with retarding forces, affect the separation.

The Retarding Force (absorptive effect) of the material on the thin layer sheet works by attracting the substance to the surface of the particles of the silica gel. In contrast, absorption refers to one substance's entering into the body of another (e.g., water into a sponge). The molecules of the substance adhere to the surface of the silica gel. The substance spotted will be absorbed by the particles of the layer. Absorption is reversible and the gel will gradually release most substances back into the solvent as it flows over the spot. Absorption is stronger for some substances than others, so release of substances from the spot will vary from substance to substance. This helps separation. As the solvent flows, the more strongly absorbed materials will be held back while the less strongly absorbed will move ahead of them.

The proper choice of solvent in reference to solubility and absorptive effects of the thin layer of silica gel on the sheet will determine the ease of separation of substances in the spot. The retarding force of the silica gel works with the propelling force to case the separation of the mixture in the applied spot.

IDENTIFICATION OF WRITING INKS

Ballpoint, Fountain Pens and Felt Tip Pens

Inks have been around since ancient times, almost as long as writing. They date back to 3000 B.C. when the first inks consisted of finely divided charcoal suspender in water—the forerunner of the India or China inks. There are many brands of ink available today. These are limited to the availability of suitable dyes. In one laboratory, one investigator completed a spectrophotometric examination of 238 ballpoint pens and 100 different dyes. Since there are so many different dyes and inks available, there has been a need for rapid screening for pinpointing suspected documents and/or handwriting.

Fortunately, most pens used today are of the ballpoint type. This tends to narrow the search somewhat. Fountain pens appear to be in decreasing use—replaced by the felt tipped type of pen. These three general types of pens will be describe in the following directive.

It is perhaps auspicious that very seldom has there been the manufacture of a pure dye. Nearly all dyes, although they appear to be of a single color, will separate into several colors upon chromatography. On this property is based the identification procedures made possible by the use of the MQDA500 kit. The chromatogram obtained is a fingerprint of the ink.

Questioned document examined with No. 782ADC Tiger Twin Dual Wavelength (shortwave/longwave) UV light source and No. 316M Magnifier. Both are supplied with the MQDA500 Kit.

PRELIMINARY PROCEDURES

There are a number of examinations that should be carried out before chemical examination of a document.

1. First, photograph the sheet and record its appearance.
2. Record the appearance to the eye in normal and reflected visible and ultraviolet light. This can sometimes give information as to the type of pen used. It will also show erasures and disturbance of fibers and breaking of their size in the paper. It will show dis-

colorations from chemical erasure. Under ultraviolet light, some inks (particularly those other than black or blue in color) will have a characteristic fluorescence. Many red inks contain Rhodamine B which is highly fluorescent.

3. Examine the document under a hand lens or microscope. This sometimes gives clues to which type of pen was used. Ballpoints, since they require pressure, can sometimes be pinpointed. They generally give an uneven colored surface. Often a thin dark line can be seen in the middle of the ink line. Felt Tipped pens, because of more even contact with the paper surface, give a more uniform colored line. Both fountain pens and felt tipped pens penetrate the paper better because of their solvency. This may also result in more ragged edges of the ink line.
4. Examine the document under a series of colored filters in visible light, alternate light sources and ultraviolet lights. Characteristic colors or fluorescence can be more easily seen sometimes.
5. Apply a drop of water to a section of the writing. This may differentiate between fountain and ballpoint inks. It will differentiate carbon black inks or India inks that smear at once and can easily be washed off or even lifted with a blotter. Ballpoint inks will generally show no effect.

Thin Layer Chromatography

After the physical examinations are completed, the chemical characteristics of the ink may be attempted. Careful execution of these tests is necessary for satisfactory results. Remember, a little bit of color goes a long way. Much information can be obtained from a small section of writing on paper. Even a single letter can provide enough ink to test. Think “micro” in these tests and your results will be surprising. The equipment supplied will make it easier for you to do these tests.

TEST PROCEDURES

1. Using one of the cotton swab lifters (No. KCP210) dipped in the No. ERDS4D Extract Reagent for Document Sampling, transfer a small amount of ink from the document to the center of the line found on the Thin Layer Chromatographic Sheet (No. KCP215). Touch the sheet lightly—it is better to apply 3 or 4 dabs to the sheet than to apply a large spot. Apply the spots rapidly to prevent evaporation of the solvent in the swab. Allow the spots to dry.
2. Place Mobile Solvent A (No. TLC001A) in the bottom of a glass jar marked “Mobile Phase Developing Jar”.

Now place the chromatographic strip in the jar. Replace the cap and notice that the spot should be just above the level of the solvent. Allow the solvent to travel almost to the top of the sheet or 1/4" (6mm) from the end. Place the jar on a flat surface—DO NOT agitate.

Observe the separation of colors in the ink (fountain pen inks and nearly all felt tipped pen inks may show no movement of colors—however, one or two felt tipped pen inks may show some movement). The ballpoint inks will all have some separation of color in this procedure. Examine visible color and invisible color under ultraviolet light. The ballpoint inks that show some colors will have characteristics that can be used for identification. The number and color of spots should be recorded. *NOTE: Both visible and ultraviolet light examinations must be carried out.*

3. Repeat the procedure using Mobile Solvent B (No. TLC001B) in a clean Mobile Phase Developing Jar. All inks, except carbon inks, will show separation of colors when subjected to chromatography with this solvent. The number of colored spots, in both visible and ultraviolet light, can be very characteristic of the ink and source of the ink. Not all inks will show fluorescence when examined under the ultraviolet lamp. This further serves to narrow down the search. Even black ink will separate into a wide variety of colors.
4. Repeat the examination of the lifted ink using Mobile Solvent C (No. TLC001C) and a clean Mobile Phase Developing Jar. The greatest separation will be found here—more spots will separate from an individual ink. Felt tipped pens usually have fewer separated colors and fountain pen inks will have the largest number of separated zones. The ballpoint inks are intermediate between these two in number of spots. The contrast between the different inks will be so pronounced that it may be possible to identify the brand as well as type of pen used.
5. For preparation of reference chromatograms to compare with the suspected ink, known brands, types and colors of inks must be examined. This is facilitated somewhat by the deductions of the above experiments, both color and visual examinations.

NOTE: Dispose of all used reagents in accordance with all Federal, State and Local regulations.

REFERENCE CHROMATOGRAMS

1. To prepare the chromatogram from the felt tipped pens, lightly touch the tip to the center of the line found on the

thin layer sheet. The absorbent sheet will remove the ink from the tip. Notice how easily the ink flows from the tip to the sheet. Remember to use very little since large spots do not separate well. It is helpful sometimes, however, to start with a small spot and then repeat with a larger spot to see dyes which are present in small amounts. Separate in the desired solutions and compare.

2. To prepare the reference comparative chromatogram of fountain pen inks, dip a cotton swab with the ink and touch lightly to the white side of the thin layer sheet and separate in the solutions used before. Compare with the suspect ink as to number of spots and color in both visible and ultraviolet lights.
3. Ballpoint inks do not flow well and are not as easily removed as felt tipped or fountain inks. Nevertheless, ink can be removed from the cartridge pen in the following manner. Remove the cartridge from the pen and dip it down into the small vial marked "Extract Reagents—Ballpoints". Leave it in the solution until enough color is removed. Sometimes the point has to be pushed and wiggled around on the bottom of the tube to get the ink to flow out. Failing that, it is possible to cut off the top of the cartridge and remove some ink with the lifting swab. With the lifting swab, transfer some of the colored solution to the thin layer sheet as previously described. Again, compare with the suspected ink. Be sure to apply a minimal amount of ink to the thin layer sheet. It is easier to obtain separations when the spot is not overloaded with ink when it is applied to the chromatogram.

Obtainable Points of Comparison

- *Number of spots*
- *Color of spots*
- *Mobility of spots*
- *Color when exposed to ultraviolet light*
- *Fluorescent or non-fluorescent spots in the separated ink*
- *Mobilities in Solution B as compared to Solution C will differentiate ballpoint pen ink from a felt tipped or fountain pen ink*
- *Mobilities in three different solutions*

VALIDITY

The colors and number of colors of the various inks are very characteristic. Rarely will the inks from two different manufacturers match. There may be some spots similar, but there are others not matching. After lifting ink from a document, it should be possible to match it with a known brand of ink from a suspect. By the same token, it is pos-

sible to show no match.

Sometimes it is necessary to apply a sample of the suspected ink to a piece of paper the same as the suspected document. From this, a spot is applied to the thin layer sheet and developed in the solvent. More precise comparison can be made this way. Sometimes, for example, an ink that is applied directly to the thin layer will show five separate zones, while (after removal from the paper) the same ink may show only four zones. The reason for this is that the paper had retained one of the dye components of the ink.

To measure mobilities of spots on the chromatograms for the record, mark the center of the spot. Mark the solvent front—this should be done as soon as the sheet is removed from the jar. Then measure in millimeters the distance from the point of application of the spot to the solvent front (SF). Measure the distance from the point of application to the center of the spot. The ratio (Rf) with respect to distance traveled by the solvent or

$$\frac{\text{distance to spot}}{\text{distance to solvent front}}$$

gives a measure of mobility that is universally used.

As with any method used in the laboratory, the investigator should get some experience with manipulation of the test described before attempting a test on a suspected document. The simplicity and ease of thin layer chromatography will be learned rapidly with practice. Several types of pens of different colors should be obtained and observations made of the dye separations. The test chromatograms can be labeled as to type of pen, color of ink, and manufacturer and filed in order to build up a library of chromatograms for future comparisons.

INK COMPARISON REAGENTS (IR1-IR10)

One of the most frequent problems encountered by the examiner of questioned documents is that of ink comparison or identification. The problem usually is to determine whether or not the original writing has been tampered with in any way or if new writing in has been added—either at the end of a paragraph or even on an additional page.

The main purpose of this collection of Ink Comparison Reagents is to enable the examiner, by observation and experimentation, to be able to make a definite statement or draw a definite conclusion to his suspicions regarding the

questioned document. The ten reagents supplied with your kit are ones that will produce the most varied reaction on modern writings that are procurable on the commercial market.

Use of Ink Comparison Charts

Before testing the actual questioned document, the examiner should choose similar types of inks and experiment with them on the Ink Comparison Chart (see sample on back page). This is done by placing a sample of the inks from one particular source or pen throughout the ten spaces provided on the horizontal lines. This will allow the examiner to become very familiar with the reactions of the particular color of ink he is interested in by observing the reactions of the inks in relationship to the reagents. Due to unpredictable atmospheric changes, size of the pen, quality of paper and other factors, certain things such as color and the reaction time may vary slightly. However, this should not prevent the examiner from obtaining good ideas of what to expect during his examination. Naturally, if the basic ingredients of the inks are the same, the reactions will vary very slightly—but, if careful observation is made with the microscope, a noticeable difference can be seen.

Procedure

After the reagent is chosen, apply it to a thick stroke of writing with the applicator supplied in the cap of the vial. The lease amount of reagent possible should be applied in order not to deface the document more than necessary. The reactions or any changes in color should be carefully noted. Changes may occur as soon as five minutes after application of the reagent depending on the composition of the ink. Ordinarily, this is long enough to be able to draw a conclusion as to whether or not the inks are from the same source or different ones. Decisions such as these, however, are up to the examiner to make.

After the reactions have been noted, the reagents may be washed with distilled water (this should be done with a cotton swab). Dip the swab in the water and brush it over the area. If there is excess reagent on the paper, it should be blotted before the application of water. This will stop any further reaction of the ink on the paper.

OTHER METHODS OF QUESTIONED DOCUMENT ANALYSIS

For more detailed information on Developing Erased Inks, Indented Writing Restoration, and use of Questioned Document Analysis Plates, refer to their individual Technical Information Sheets included in your kit.

PRECAUTIONS: UV RADIATION

The three areas of ultraviolet radiation are UV-C at 100 to 280nm, UV-B at 280 to 315nm, and UV-A at 315 to 400nm. UV-C is the shortest wave ultraviolet radiation and UV-A is the longest wave ultraviolet radiation.

The retina of the eye is not very vulnerable in the ultraviolet or the far-infrared portions of the spectrum. It is the cornea and the lens that absorb ultraviolet. High exposure levels can permanently damage these structures of the eye. Intermediate levels in the UV (200-320nm) cause greater injury to the cornea, which is severe but temporary. The injury, photokeratitis, may last for only one or two days but is extremely painful. Near-ultraviolet (long wavelength UV-A) is absorbed heavily in the lens of the eye. Damage to this area of the eye may not be evident for many years and may have lasting effects.

Human skin is also susceptible to radiation injury. This susceptibility occurs in the range of radiant energy present in the ultraviolet spectral region of 200-320nm. This type of radiation can cause severe sunburn. Certain photosensitizing chemicals greatly increase the sensitivity of the skin. Previous exposures to specific wavelength bands that are generally in the long wavelength ultraviolet and visible portion of the spectrum also sensitize the skin. Some orally administered drugs such as tetracyclines and common pain relievers also cause photosensitization.

The factors predisposing individuals to possible harm from ultraviolet radiation are:

- Sensitivity of the individual
- The length of exposure
- Intensity of the ultraviolet light source
- Light source/surface distance

Recommended Personal Protective Equipment:

- UV absorbing face shield or glasses with side shields
- Long sleeved laboratory coat or overalls
- Opaque cotton or garamid fiber gloves

SIRCHIE shortwave UV lamps utilize low-pressure mercury lamps, which emit radiation in the UV-C (254nm) spectrum. Any amount of exposure to these lamps should be considered hazardous and protective equipment for the eyes and exposed skin must be worn. When using any UV lamp, avoid needless exposure to radiation and turn the lamp off when not in use.

INK COMPARISON CHART FOR EXPERIMENTAL TESTS

Instructions:

The chart provided on the next page will enable you to experiment with actual specimens of writing inks. The vertical columns from #1 to #10 inclusive are provided so that tests with our Ink Comparison Reagents can be made systematically. These experiments will familiarize you with the reactions of each reagent when applied to whatever specimens of ink you have chosen.

The reagent should be applied to the ink specimen with separate applicators. Since each reagent has its own applicator in the cap of
the vial, cross contamination is eliminated. Dip the applicator brush into the vial of reagent and make a downward stroke across the specimen of ink. After making this stroke, immediately dip the applicator into a small jar of water or alcohol and wipe it clean with a cloth. This cleansing is necessary to prevent any of the inks from becoming mixed with the reagents.

Starting with #1 Reagent, apply the reagent to each specimen of ink in the vertical column. Any reaction which takes place can be observed and a notation in pencil can be made in the space directly beneath each writing specimen. Follow the same procedure for the remaining nine reagents.

REAGENT	#1	#2	#3	#4	#5	#6	#7	#8	#9	#10
REACTION										
REACTION										
REACTION										
REACTION										
REACTION										
REACTION										

MQDA500 CONTENTS:

- 1- 214C2 Iodine Print Fixative, 2 oz. (59ml)
- 1- 215CS2 Iodine Print Fixative Remover, 2 oz. (59ml)
- 10- 288DP Disposable Pipettes w/Capillary and 3ml Bulb
- 1- 316M Magnifier, 2.5X, 2.5" (6.3cm) dia.
- 1- 374H Questioned Document Plate Combination, Set of 4
- 1- 782ADC Tiger Twin Dual Longwave/Shortwave UV Light Source, 6V DC
- 1- ACN2 Acid Neutralizer, 2 oz. (59ml)
- 1- AMP2066 Iodette Ampoule, 3g, 6 ea.
- 1- DISW2 Bottles of Distilled Water, 2 oz. (59ml)
- 4- EINK1 Erased Ink #1, 2ml vial
- 4- EINK2 Erased Ink #2, 2ml vial
- 4- EINK3 Erased Ink #3, 2ml vial
- 4- ERBP01 Extract Reagents for Ballpoint Ink
- 1- ERDS4D Extract Reagent for Document Sampling
- 4- ERFT1 Extract Reagents, Felt Tip Ink
- 4- IR1 Ink Reagents No. 1
- 4- IR2 Ink Reagents No. 2
- 4- IR3 Ink Reagents No. 3
- 4- IR4 Ink Reagents No. 4
- 4- IR5 Ink Reagents No. 5
- 4- IR6 Ink Reagents No. 6
- 4- IR7 Ink Reagents No. 7
- 4- IR8 Ink Reagents No. 8
- 4- IR9 Ink Reagents No. 9
- 4- IR10 Ink Reagents No. 10
- 2- IRS100 Indentation Removal Solutions, 1 oz. (30ml)
- 4- ISS3 Indentation Solutions, Strong
- 4- ISW1 Indentation Solutions, Weak
- 1- KCP139 Tweezers, Plastic, 4.75" (12.1cm)
- 7- KCP154 Pipettes w/Suction Bulb
- 1- KCP166 Comparator w/Reticles
- 20- KCP167 Alligator Clips
- 10- KCP210 Swabs
- 10- KCP215 Thin Layer Chromatography Sheets, 1" x 3" (2.5cm x 7.6cm)
- 24- KCP217 Cotton Balls
- 15- KCP227 Filter Papers, 1" x 2" (2.5cm x 5.1cm)
- 4- KCP228 Glass Jars w/Closure, 4 oz. (118ml)
- 4- KCP233 C-Cell Batteries, Alkaline
- 1- PFP200 Fingerprint Magnifier, Model M2
- 1- SF00771 Latex Powdered Glove Pair, Ambidextrous, .005" Thick, Large
- 4- TLC001A Mobile Solvent A
- 4- TLC001B Mobile Solvent B
- 4- TLC001C Mobile Solvent C
- 10- ZTE021 Iodette Fuming Bags, 9" x 12" (22.9cm x 30.5cm)
- 68- KCP193 Brush, Applicator 3.25" (8.2cm)
- 1- MQDA5001 High-Impact, Copolymer Carrying Case w/Handle; Dimensions: 18" x 13" x 14.5" (45.7cm x 33cm x 36.8cm); Weight: 19.7 lbs. (8.9kg)