

A NEW TECHNIQUE FOR MEASURING WATER UPTAKE OF
LITHOGRAPHIC INKS:

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ABSTRACT

A new technique for measuring water uptake of lithographic news inks is described. The new "titration" test is an automated approach which is quick and reproducible. Water uptake is determined by monitoring torque changes on a mixing blade as fountain solution/water is flowed into mixing ink. The torque profile is characterized by two parameters, Emulsification Capacity (EC) and Change in Torque (ΔT). The EC parameter is a water uptake value in grams of fountain solution emulsified/100 grams of ink. The ΔT parameter reflects the nature of rheology during emulsification.

The utilization of the new technique on lithographic newspaper inks is presented. Comparison to the Surland approach (via Duke Tester) has been made. Examples of quality control and formulation applications are described.

INTRODUCTION

Lithographic press performance is dependent on establishing proper ink/water balance. The control and measurement of water uptake by an ink are important aspects of lithographic ink formulation. The measurement of the water pick-up is a valuable quality control parameter. Among the various methods have been the so-called "mixmaster" approaches (Surland 1980, 1983; Fetsko 1980; ASTM D4942; NPIRI Task Force 1990)

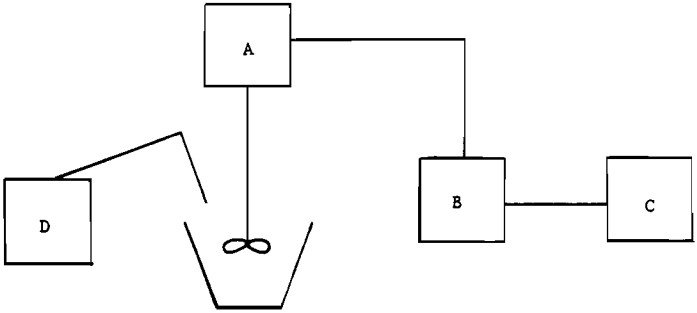
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and variations of the Litho Break Tester (Tasker et al. 1990; Irg 1985; Van Esch 1990). The original work on the "mixmaster" approach by Surland (1980, 1983) was an attempt to quantify water uptake via a simple laboratory method and correlate the data to press performance. Although there have been many debates concerning the validity and utility of the method (Tasker et al. 1989), the technique does allow relative laboratory comparison of lithographic inks as much as relative viscosity and other physical measurements do. The extrapolation of the measurement to press performance is as limited as any single measurement might be. The "mixmaster" methodology has recently been standardized and an ASTM test method established. The resultant ASTM tests, D4942A and D4942B, utilize the commercially available Duke tester. ASTM D4942A is a single point, five minute water uptake measurement; whereas D4942B represents a refinement of Surland ten point, ten minute test procedure. The latter method can be heavily dependent on operator skill and is rather time consuming. If the objective is to monitor water uptake for quality control purpose, we describe a new procedure which is suitable as a quality control and formulation tool. It is quick, reproducible and provides new data regarding emulsion rheology. The test method is automated to provide a direct hard copy of the results. Examples are provided of the utility of the new technique, termed "titration test", as quality control tool in production environments, and as an aid in formulation to improve press performance.

EXPERIMENTAL

Figure 1 is a schematic representation of the test equipment. The torque sensing stirrer (G.K. Heller HST10-NMEV) has a torque range of 1600 gm-cm. Changes in torque are monitored via output to a strip chart recorder. Constant speed is maintained throughout the measurement time. The work described in this paper, utilizing newspaper inks, was run at 1200 rpm. The sample (25 gms ink) is placed in a four ounce tin can and seated in a holder which can be thermostatted to 30°C. For mixing, a standard

flat propeller with 3 blades is used. The blade is displaced 0.5mm from the bottom of the ink receptacle. The titration of water or fountain solution is conducted via a 10 rpm peristaltic pump fitted with a 25 gauge needle for dispensing into the mixing ink at a flow rate of 0.07 gms/ sec. The output results from monitoring torque changes during the water addition to the mixing ink.



- A - TORQUE SENSING STIRRER
- B - SPEED CONTROLLER
- C - TORQUE OUTPUT - STRIP CHART RECORDER
- D - PERISTALTIC PUMP FOR FLUID ADDITION

Figure 1 - Diagram of Test Method

RESULTS AND DISCUSSIONS

The design of this technique was the result of observations that a distinct endpoint could be reached when titrating an ink with water or fountain solution. The endpoint is characterized by an excessive turbulence and slippage on mixing blade when the ink sample no longer accepts the added water. At this point, two phases exist, resulting in the observed mixing patterns. The new apparatus measures the rheological changes via the torque sensing stirrer. Figure 2 shows a typical torque vs. time response. Initially, the torque level represents the ink rheology under the temperature and mixing conditions. The addition of water or fountain solution produces a profile in which torque increases as the emulsification proceeds and then will reach a metastable region where torque

changes erratically but still appears to be emulsifying under these shear conditions. Eventually the torque drops below the initial level. The full realization of two phases causes the ink to recede from the mixing blade, resulting in the reduction in torque. The effects described here under these conditions are not unlike the behavior observed for emulsions examined under the shear conditions of a cone/plate rheometer (Chou and Cher, 1989). The shear and temperature conditions will dictate when emulsion capacity is reached and thus not allow stability with additional water. The metastable region most likely reflects that coalescence of droplets is beginning to occur. We have found that the most reproducible way to characterize the emulsion capacity (EC) is the amount of liquid added between initial starting torque level and the point where the torque goes below this initial torque indicating excessive slippage. (See Figure 2). Using the time axis to calculate the amount of liquid added, the emulsion capacity can be defined in units of grams water or grams fountain solution per 100 grams ink, similar to the Surland emulsification scale.

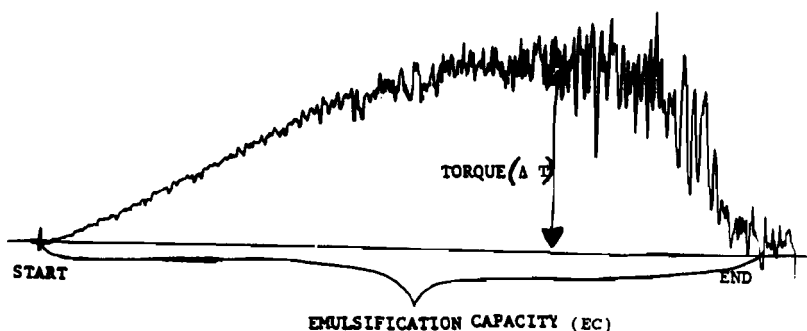


Figure 2 - A Typical Torque vs Time Profile

In addition to the emulsification capacity, the profile is characterized by the quantity, ΔT , which represents the total change in torque from start to maximum level. This value is given in relative terms in millivolts relative to 1V full scale. The actual torque values can be obtained in gm-cm units if desired. The larger the value

of ΔT , the greater the rheological change (i.e. viscosity and yield) occurring during emulsification. Excessive changes can aggravate press problems such as ink "boiling" over the rail. The emulsion rheology has received greater attention recently with regard to its effects on emulsion stability and printability (Bassemir and Shubert, 1985; Chou and Baun, 1989). As will be noted below, this new parameter can be an important formulation aid.

The emulsification capacity (EC) reflects the maximum uptake under the chosen conditions. The conditions described here, as are suitable for newspaper inks, represent a mixing shear rate of approximately 210 sec^{-1} . The effect of changing shear rate by changing speed is shown in Table 1. Sufficient mixing must occur before a constant emulsion capacity results at the chosen flow rate. The lack of adequate mixing causes a two-phase system to arise immediately. The ΔT parameter was noted to decrease as shear rate increased. This is expected if emulsion rheology is altered by shear rate. The test provides for shear rate and mixing conditions to handle different viscosities of news inks equally well. For example, conversion offset, keyless, open fountain and standard injector viscosity inks, can be easily examined via the titration test. In addition, the overall torque sensing unit can be scaled so as to handle web offset/heatset inks.

Table 1 - Effect of Shear Rate

<u>RPM</u>	<u>Shear Rate (sec^{-1})</u>	<u>EC</u>	<u>ΔT</u>
250	42	Insufficient Mixing	
750	125	Erratic Mixing	
1250	210	80	250
1750	292	79	230
2500	420	78	220

The effects of flow rate and temperature were examined. The chosen flow rate was established arbitrarily (i.e. due to pump considerations) as a benefit to insure adequate mixing time during addition. The conditions chosen reflect the relative mixing and emulsification kinetics for news inks and thus for another class of lithographic inks, a different set of parameters may be necessary. There was only a minimal effect on EC and ΔT over a range of 20-40°C.

There are other features of the output profile which reflect the characteristics of emulsification, but have yet to be deciphered. The band width during titration represents a relative rate of emulsification. A more receptive ink provides a smoother curve with less change in band width. At this time, we have not been able to satisfactorily correlate this property with other measurements. Another aspect yet to be fully understood is the nature of metastable segments prior to the endpoint. The transitions can be gradual or extremely abrupt, depending upon the chemical composition.

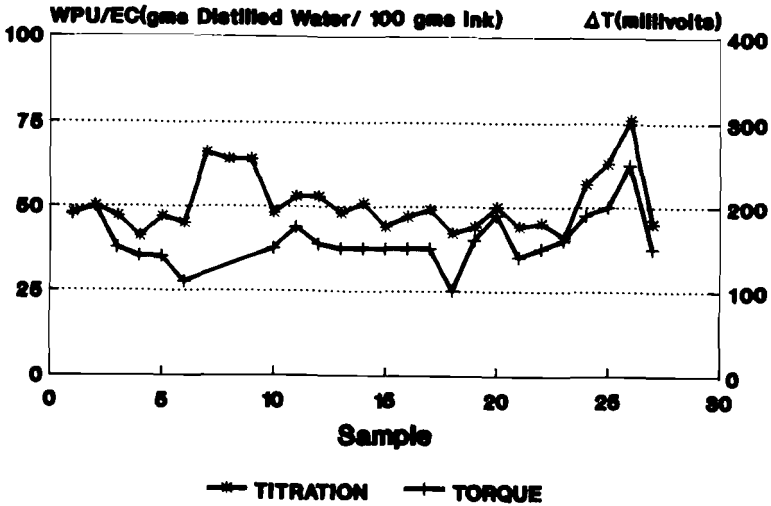
Our initial interest in this technique centered around its use as a quality control tool for manufacturing sites. A ten point, ten minute Surland curve (via Duke Tester) was being gathered on individual batches of ink. The intent of these measurements was to monitor batch to batch variation in emulsification at ten minutes. Table 2 contrasts the titration approach with this method. As can be seen, a significant time savings and an improvement in reproducibility could be realized.

Table 2 - Comparison of Titration to Surland

	<u>SURLAND*</u>	<u>TITRATION</u>
Shear Regime (1/s)	Low	Moderate
Ink Volume (gms)	50	25
Data Obtained	WPU	WPU
	Emulsification	Emulsion
	Rate	Rheology
Temperature Control	None	30°C
Measurement Time(min)	30-40	5-8
Reproducibility	Good	Excellent
*Ten point, ten minute test using Duke Tester		

The technique also offers a competitive alternative to a five minute single point measurement.

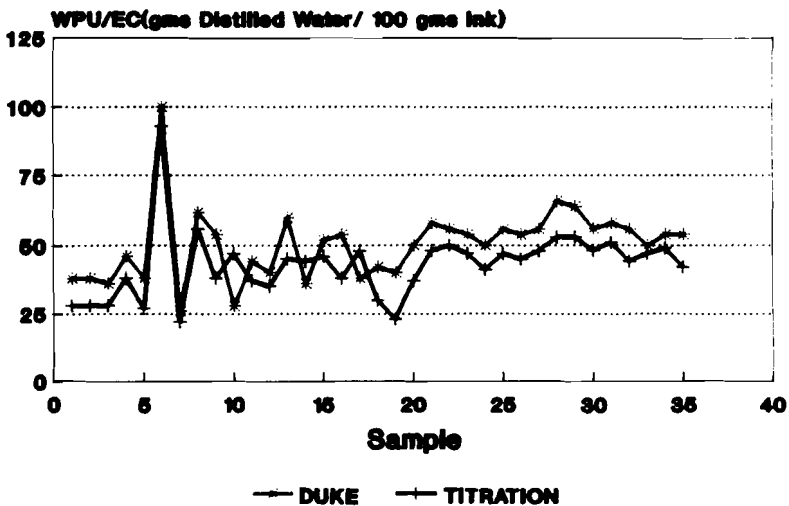
Figures 3-5 are representative data obtained from a series of production batches of black news inks. There is generally a close correlation under our given conditions. In addition, the new information from the ΔT data can be useful in assessing fluctuations in raw material properties.



Open Fountain Black News Ink

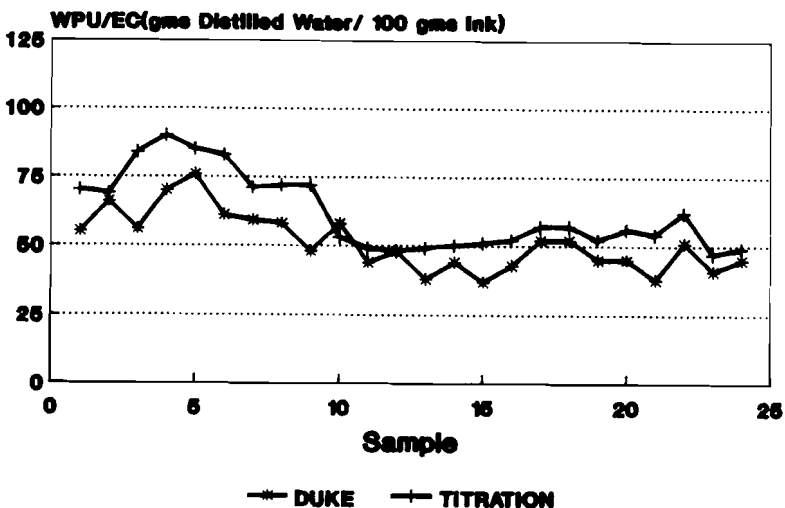
Figure 3 - Comparison of Titration EC Value With Ten Minute Point From Duke Test. (Open Fountain Black)

This technique does not easily manifest the rate of uptake, but does provide a quick, reproducible water uptake value. As noted previously, ΔT is a new parameter which can be utilized in quality control or more effectively in formulating the lithographic inks.



Open Fountain Black News Ink

Figure 4 - Comparison of Titration EC Value With Ten Minute Point From Duke Test. (Injector Black)



Injector Viscosity Black News Ink

Figure 5 - EC and ΔT for Black News Ink.

Figure 6 shows an example of torque changes due to minor alterations in formulation.



Figure 6 - Comparison of Two Black News Ink Formulations Where One Component Substituted at Equal Level With Different Acid Values.

A - EC=78, $\Delta T=210$

B - EC=48, $\Delta T=190$

Figure 7 shows an example of the effect of changing fountain solution on the profile obtained. Thus, the parameter appears to be sensitive to the nature of surface active materials which contribute to emulsion properties and yields additional data regarding the nature of emulsified inks.

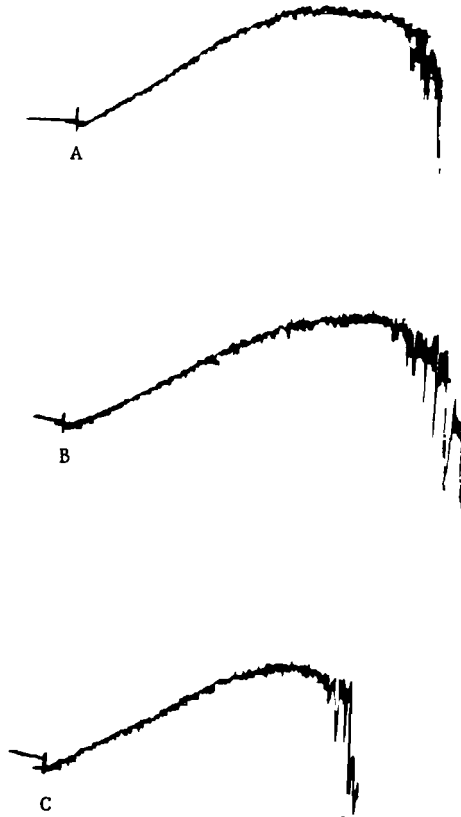


Figure 7 - Effects of Fountain Solution on EC and ΔT .

- A - Alkaline Solution 1: EC=58, $\Delta T=280$
- B - Neutral Solution: EC=64, $\Delta T=250$
- C - Alkaline Solution 2: EC=48, $\Delta T=230$

SUMMARY

The new technique described here represents a quick, reproducible method to assess water uptake and emulsion rheology. The test is automated in such a way to allow greater control of measurement conditions and insures easier operation than "mix-master" methods. In addition to its utility in quality control, formulation of inks can be guided from the data obtained. The adaptability of the technique to other lithographic inks (i.e. heatset) is under study. Future work will focus in deciphering emulsification rate via this type of measurement.

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