

Quick and Easy Way to Characterize Low Voltage (80-125 kV) EB Accelerators Using Fast Check Strips

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Abstract:

With the increased use of low voltage Electron Beam accelerators for curing coatings, inks, laminating adhesives and cross-linking films for packaging applications, there is an increased demand to accurately, quickly and easily measure the required dose delivered to the product.

To meet this demand, Energy Sciences collaborated with Spectra Group Ltd. and has developed a quick and easy method to characterize the output of these accelerators accurately using EB Fast Check V2 strips. These strips are very easy to handle, read by a small color densitometer, and are fit for industrial use. They are not affected by temperature and humidity. These Fast Check strips are traceable to NIST via calibration through ASTM compliant standard dosimetry techniques. Details of the performance of these Fast Check strips in terms of repeatability, reliability in evaluating these low voltage EB accelerators will be discussed.

Introduction:

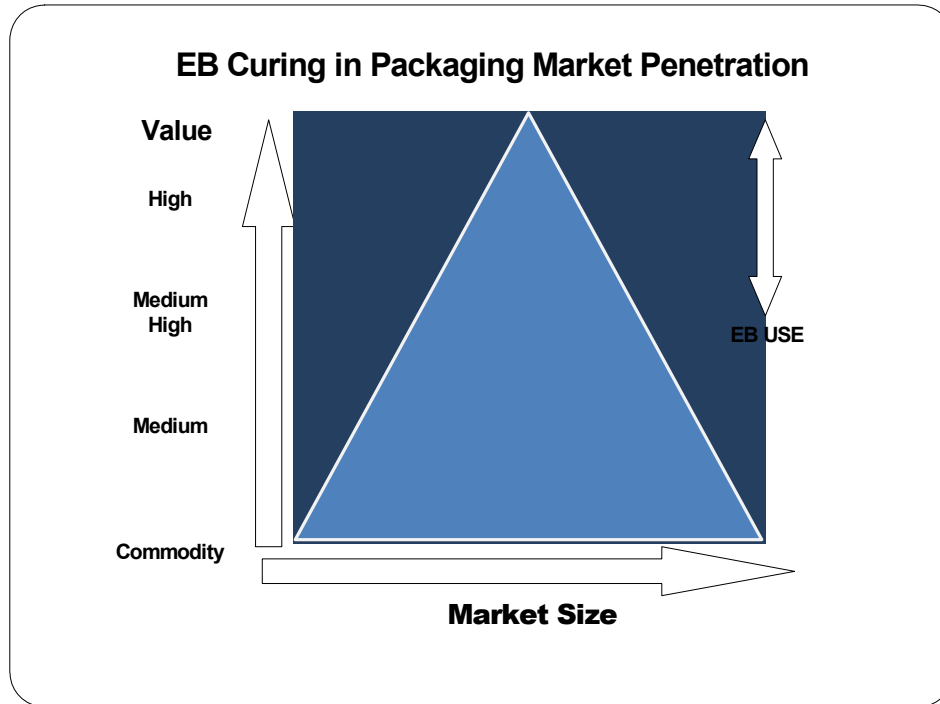
The overwhelming acceptance of low voltage low cost series of EB equipment has truly kept up with its expectation as the curing method of choice. Since its introduction in 2000 more than 125 of these type of EB systems have been sold globally processing close to 4.0 billion meter² of material annually, with greater than 90% application in packaging most particularly food packaging. In packaging these EB units are used for the following applications:

Table: 1

- **Curing of Inks**
 - **Web Offset Inks**
 - **EB Flexo Inks**
- **Curing of EB lacquers**
 - **Replacing Laminates**
- **Cross linking High barrier Shrink Films**
- **Curing of Laminating Adhesives**

Advantages of EB curing have always been known, but historically it has been limited to large converters for high value and niche applications¹. Development of low cost EB equipment increased market awareness towards lower value, higher volume applications as shown in Figure: 1. This development was further fueled by environmental mandates put forth by package sustainability, lower carbon footprint, and Cap & Trade² making energy curing in particular EB curing as the curing method of choice³.

Figure: 1



Radiation induced insitu polymerization offers significant advantages over conventional thermal based processes. One can tailor-make the desired end properties into the chemistry of energy cured materials providing a better end product economically. EB processing as shown in Table:2 has a further advantage over UV the other energy curing source, is that it can initiate the reaction without addition of photo initiators resulting in cleaner chemistry, and its output is measured by NIST traceable dosimetry techniques⁴.

Table: 2 Advantages of EB Curing

- **NO VOC, Lowest Carbon Foot Print Curing Option**
- **Lowest Migration odor and taint. Food packaging compliant**
- **Cleaner Chemistry, No Photo-initiators or peroxides or other additives**
- **Highest degree cure results in unsurpassed end properties like**

- **Gloss**
- **Controlled COF**
- **Abrasion resistance**
- **Good Quality Control through NIST traceable dosimetry techniques and closed loop electronics.**

As the market acceptance of EB processing is increasing. These EB equipment are being used at converter sites all over the world usually in 24/7 type of operations where it is absolutely mandatory to minimize downtime. At the same time it is absolutely mandatory to check the output of these EB machines especially after routine maintenance to ensure that the product receives the required dose to achieve the desired cure level.









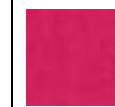
NIST traceable FWT nylon radiochromic dye dosimetry techniques work fine but unfortunately do not provide the capability of on-site testing of the EB units situated at various locations around the world. Usually these dosimeters need to be shipped back to a laboratory having controlled conditions to be analyzed, which can take in some cases 2-3 days. As mentioned earlier the downtime associated with this technique could be detrimental to the high volume processing requirements.

To achieve a quick reliable indication that the EB unit is functional EB Fast Check Strips have been developed and can be used.

EB Fast Check Strips:

EB Fast Check Strips technology is a natural extension of Spectra Group's color-on-demand expertise and the logical continuation of its thin film UV Radiometry devices (UV FastCheck and UV Intensity Strips). It was realized early on in the course of the development that not only a different color forming system had to be used to provide EB sensitivity, but also a different Strip manufacturing technique was needed as well. It turned out that printing techniques used in the production of UV Fast Check Strips did not work for the polymer matrices required for EB Fast Check Strips long shelf life and temperature/humidity independence. As a result Spectra Group used a specialty and proprietary coating method, rather than a printing technique, to apply a tightly controlled solvent based thin film onto a carefully chosen calendared paper substrate. The coating method chosen by Spectra Group allows for consistent film thickness reproduction which is key to repeatability. After solvent evaporation, the film consists of a very rigid polymer matrix, necessary for long shelf life, and EB sensitive color change system, tuned so that it has sensitivity across the wide range of administered EB dose (See Figure 2). However, for precise dose measurement the use of a handheld densitometer is strongly recommended. The color system chosen by Spectra Group does not require extensive heat or post-processing for complete color development. Room temperatures of several hours (see below) are enough to complete color formation.

Figure 2

| Dose (kGy) | 0 | 5 | 10 | 20 | 30 | 40 | 50 | 70 | 100 |
|------------|---|---|---|---|---|--|---|---|---|
| Color |  |  |  |  |  |  |  |  |  |

- 150kV, 50fpm

EB Fast Check Strips are supplied as 12"X12" inch sheets with a paper backing on permanent PSA substrate. The sheets are scribed into a matrix of ½ X ½ inch squares (total 576 squares) which can be peeled off their backing. Pre-irradiation readings (background) are taken from 15 spots on each sheet in a matrix fashion, and the average of these readings is recorded for each sheet along with the lot # and serial # from each sheet.

Reading & Calibration of EB Fast Check Strips:

Upon receiving the Spectra Fast Check strips the first step is calibration. The calibration process for Spectra dosimeters is done using Far West Technology (FWT) nylon radiochromic thin film dosimeters as a Transferred-Standard Dosimeter. The FWT dosimeters are calibrated at ESI using a dose provided by NIST (National Institute of Standards and Technology). This national laboratory subjects the films to a well-characterized gamma source (Co^{60}); this makes the calibrated Spectra dosimeters NIST traceable. The use of FWT nylon radiochromic dye dosimeters and its calibration follows the ASTM procedure.^{5,6,7}

Calibration:

Three yields (9 dosimeters on each yield) for both FWT and Spectra (See Figure 3) are run at four different doses on the ESI pilot line; this will give us a total of 108 dose points for the calibration curve.

The optical densities are read from the FWT dosimeters using a radiochromic reader and the doses are then calculated using its appropriate calibration curve generated with NIST. These doses are plotted against the direct reading of the Spectra dosimeters which are read on a Beta color reader (See Figure 4). A linear curve is then generated and used for dose calculation of the Spectra dosimeters.

Several types of curves have been generated in this calibration process, including polynomial and inverse natural log, but the linear curve is the best fit, providing us with the most accurate data when comparing dose readings (See Figure 5 for Spectra curve).

Figure 3

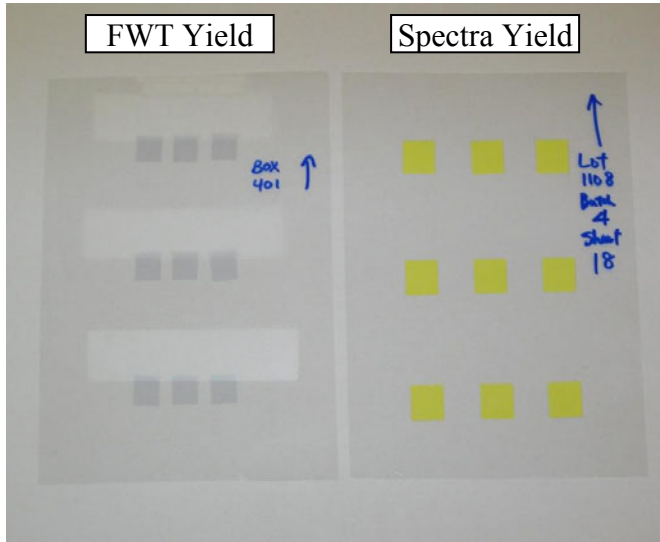


Figure 4

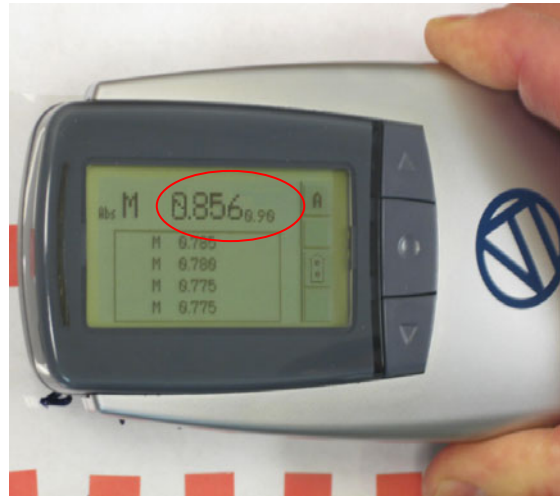
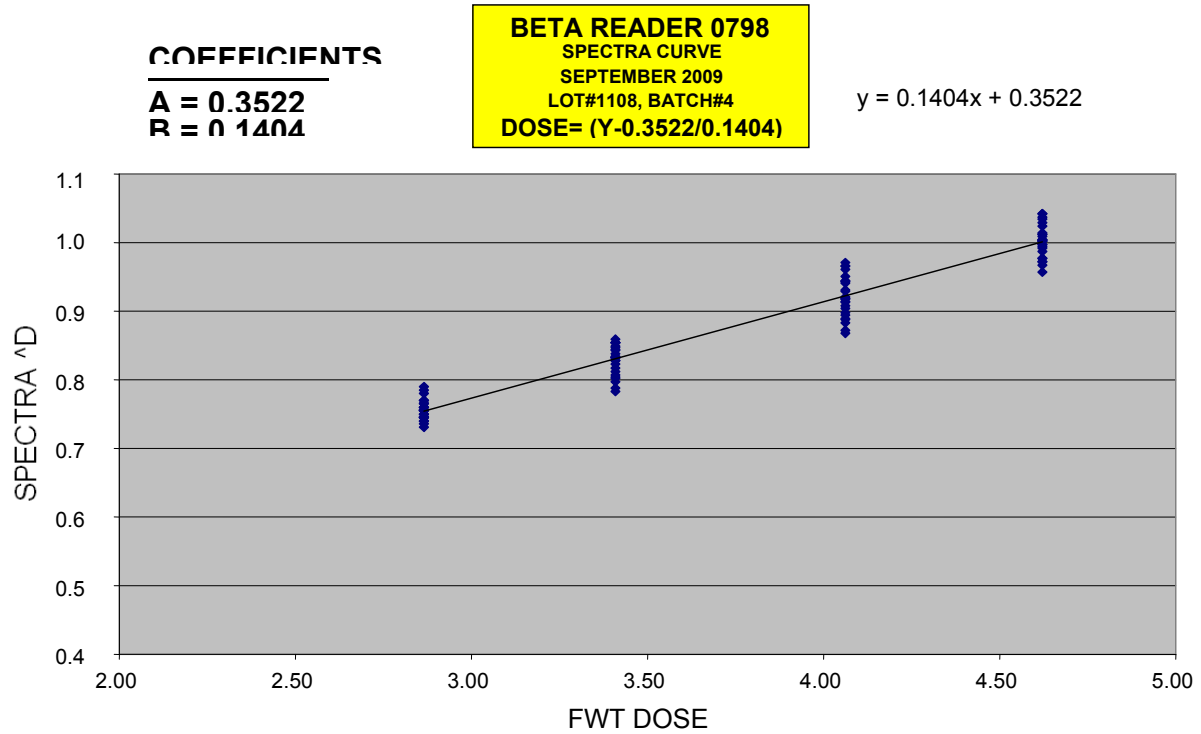


Figure 5



Reading and Recording of Spectra Dosimeters:

The Spectra chips are purchased in sticker form with a thin coating on them. They are packaged in sheets of approximate 1/2" x 1/2" squares. These dosimeters are sensitive to ultraviolet light and thus protected from UV radiation. They also must be handled with tweezers and/or latex gloves since oils from bare hands may affect readings.

Exposed dosimeter chips need to sit for four to eight hours (see development times in next section) shielded from ultra violet light before being read on a Beta Color reader, which reads the color change in the dosimeter.

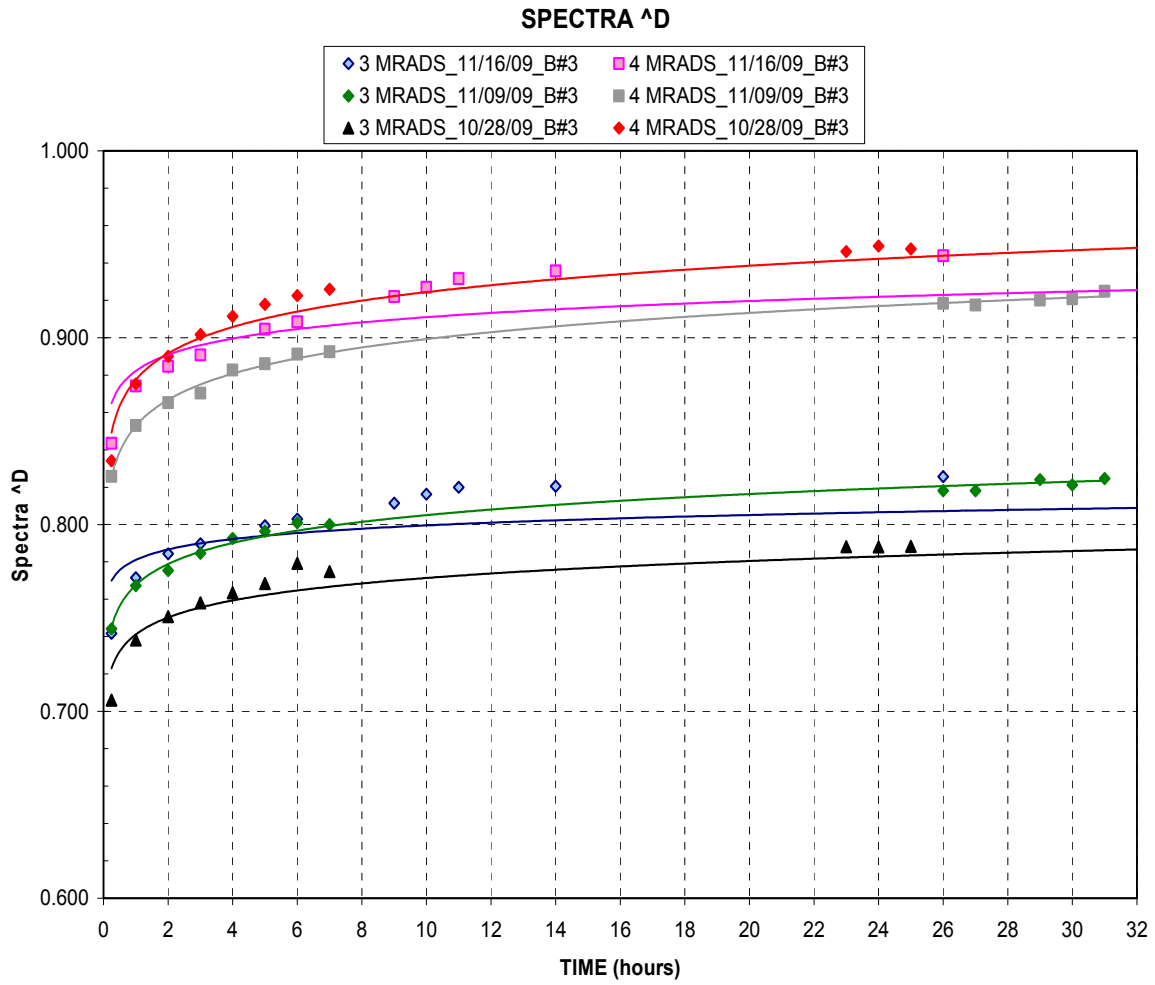
After the yields have developed, the exposed dosimeter chips are then read on the Beta reader by placing the target of the reader on top of each exposed chip. At this point the reader is pressed down carefully and a three digit number will show up (See Figure 3). This step is then repeated three times for every dosimeter read, and the average of the three numbers is documented. The number obtained is the amount of color change from the non-irradiated dosimeter to the irradiated dosimeter. This number is then entered into the appropriate dose curve equation to calculate dose.

For detailed instructions of the setup and calibration refer to the manual of the densitometer/reader⁸

Development Times:

As mentioned previously, the development time for the Spectra dosimeters is between 4-8 hours after irradiation. The dosimeters do continue to develop after the 8 hours (as shown in Figure 6), but the increase in readings is only 2-3%. Extensive data shows that consistently reading the Spectra dosimeters between the 4-8 hours development time provides us with excellent repeatability in dose readings (within the $\pm 5\%$ specification). The Spectra dosimeters do not need to be annealed, they will develop on their own at room temperature shielded from UV light. These dosimeters are not affected by temperature and humidity, unlike nylon dosimeters⁹.

Figure 6



Characterization of EB Units:

Using FWT dosimeters we characterize the EB unit for ⁴:

- Yield Measurements – these measurements are performed to measure the “K” value or the efficiency of the Electron beam.
- Depth Dose – this measures the penetration of the Electron beam into the product.
- Uniformity – this measurement is used to measure the consistency of the Electron beam over the width of the unit.

By using fast check strips we can characterize the EB unit for yields and uniformity. Table 3 shows one of the many yield comparisons we have performed. As you can see the difference in “K” values between the Far West dosimetry and the Spectra dosimetry is minimal.

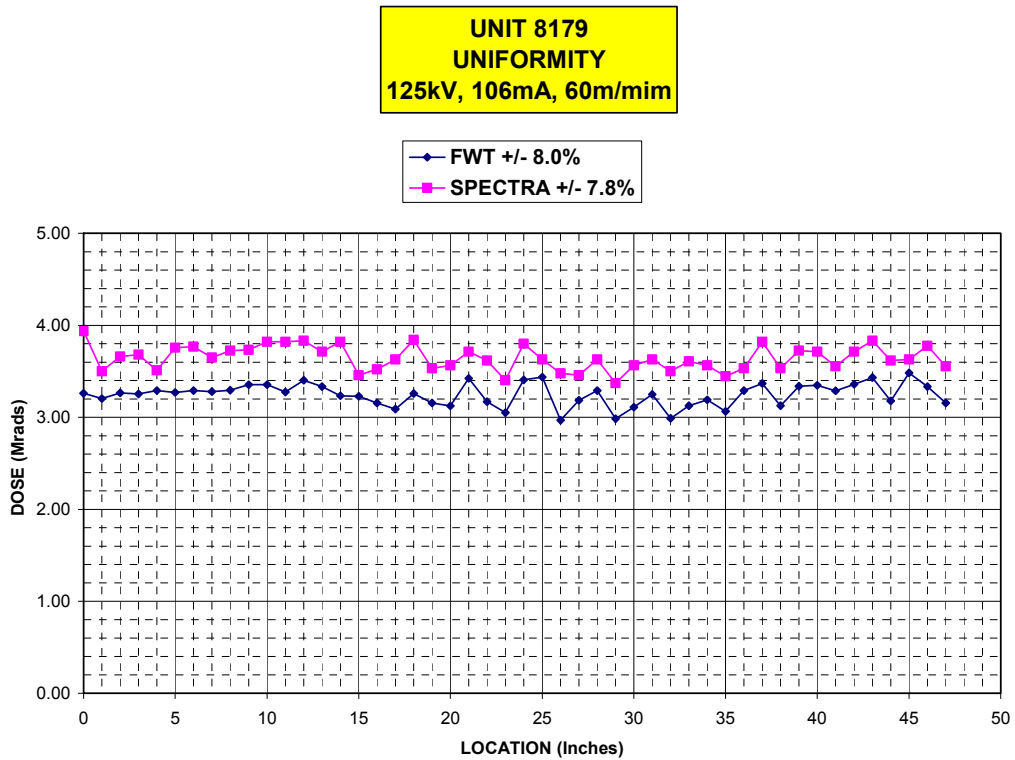
Uniformity readings can be evaluated by plotting the uniformities on top of one another to compare profiles and by using the formula $(\text{max dose} - \text{min dose}) / (\text{max dose} + \text{min dose})$, to calculate the uniformity percentage. See Figure 7 for an example of the uniformity plot.

Table 3

YIELD COMPARISON
UNIT 8201 - 125 kV
Reader #3367

| Far West Dosimetry | | | Spectra Dosimetry | | % Diff K value |
|-----------------------|----------------|--------|----------------------|--------|-------------------|
| Run | Dose Mrads | Avg. K | Dose Mrads | Avg. K | |
| Yield#1 | 3.43 | 1.86 | 3.43 | 1.86 | -0.1% |
| Yield#2 | 3.41 | 1.85 | 3.41 | 1.86 | 0.6% |
| Yield#3 | 3.48 | 1.89 | 3.44 | 1.87 | -1.2% |
| | Total Avg.K | 1.87 | Total Avg.K | 1.86 | |

Figure 7



Repeatability and Field data using EB Fast Check Strips:

We have performed numerous tests to confirm the repeatability and accuracy of Spectra dosimetry. Many of the tests were performing dose comparisons between FWT and Spectra dosimetry. Overall, the results of the testing were very good, with the majority of the results within $\pm 5\%$ of each other. All of the testing was performed on Electron Beam accelerators and many of these tests were performed at low voltage (125 kV and below) and using various different batches of Spectra dosimeters and readers.

Tables 4 and 5 show the results of some of these repeatable tests.

Table 4

SPECTRA - FWT DOSE COMPARISON UNIT EZ95 – 110KV

| 16-Mar-09 | FWT 10MICRON | | SPECTRA | | |
|---------------------|--------------|----------------|-----------|----------------|-------|
| Run | Avg. Dose | Total Std.Dev. | Avg. Dose | Total Std.Dev. | %DIFF |
| Yield #1 3 MRADS | 2.66 | 0.07 4.9% | 2.64 | 0.10 7.3% | -1.1% |
| Yield #2 4 MRADS | 4.03 | 0.07 3.6% | 3.88 | 0.16 8.2% | -3.8% |
| Yield #1 3 MRADS | 2.66 | 0.07 4.9% | 2.65 | 0.09 6.6% | -0.6% |
| Yield #2 4 MRADS | 4.03 | 0.07 3.6% | 3.88 | 0.17 8.8% | -3.8% |

Table 5

EZ 84 2/5/2009 - 110KV

10 MICRON DOSIMETERS - 3 MRADS

10 MICRON DOSIMETERS - 4 MRADS

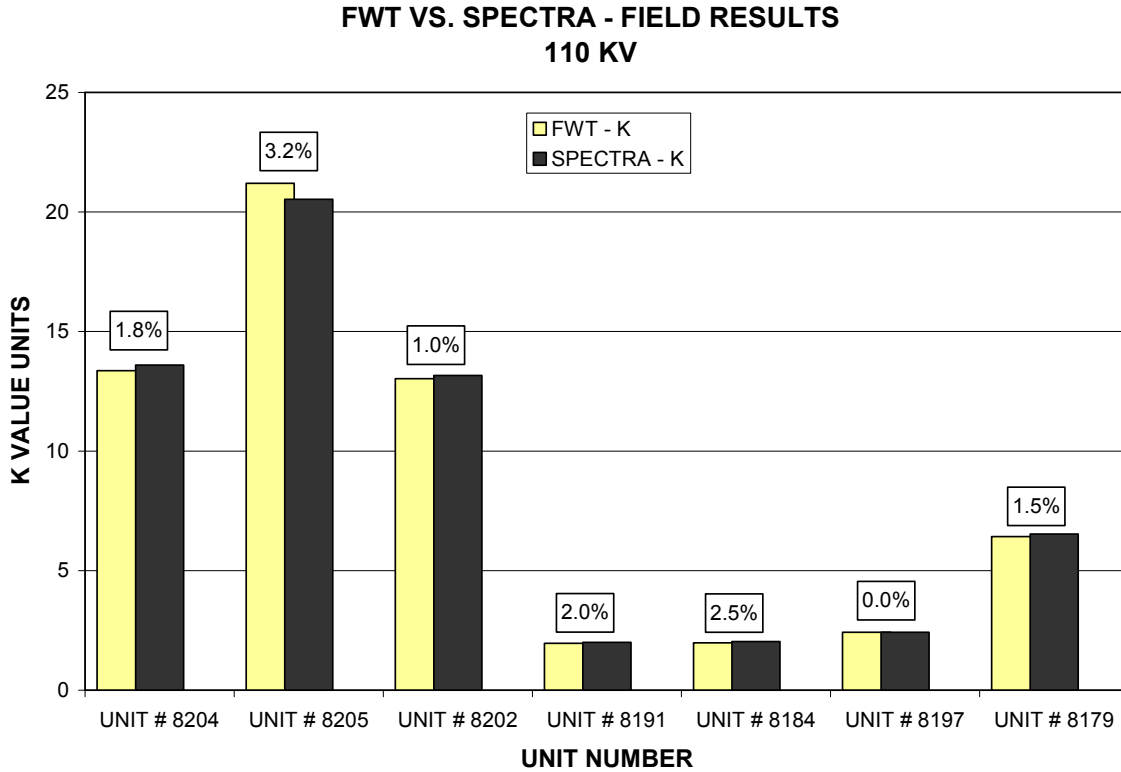
| YIELD # | FWT DOSE | SPECTRA DOSE | % DIFF |
|---------|----------|--------------|--------|
| 1 | 2.91 | 2.89 | -0.7% |
| 2 | 2.85 | 2.76 | -3.3% |
| 3 | 2.89 | 2.70 | -7.0% |
| 4 | 2.84 | 2.92 | 2.7% |
| 5 | 2.87 | 2.92 | 1.7% |
| 6 | 2.79 | 2.76 | -1.1% |
| | | AVG. | -1.3% |

| YIELD # | FWT DOSE | SPECTRA DOSE | % DIFF |
|---------|----------|--------------|--------|
| 1 | 4.02 | 4.06 | 1.0% |
| 2 | 4.05 | 3.97 | -2.0% |
| 3 | 4.01 | 3.96 | -1.3% |
| 4 | 4.05 | 3.97 | -2.0% |
| 5 | 3.97 | 3.93 | -1.0% |
| 6 | 3.91 | 3.95 | 1.0% |
| | | AVG. | -0.7% |

After numerous testing in-house as discussed above. It was decided to send these Spectra dosimeters at customer locations for post installation evaluations. Historically, our Field Service Engineers would have to run all FWT dosimetry and send it back to ESI for evaluation while waiting at a customer's site for results. This step was wasting valuable company time especially with customers in countries such as China and Brazil where shipping can be timely. The Spectra dosimetry allows the Field Service Engineers to read dosimetry, with the compact Beta reader, on site. Confirm machine performance and then run FWT dosimeters to be evaluated at controlled lab to validate machine performance as per specifications. This process has saved us days and even weeks in down time while waiting for results. Figure 8 shows Spectra dosimetry results read in the field at various locations compared to FWT results read in our dosimetry

laboratory. As can be seen at all these locations excellent inter comparison has been achieved between the two dosimetry techniques.

Figure 8



Conclusions:

Spectra dosimeters can be used to provide onsite evaluation of low voltage EB accelerators from (80 – 125kV). The result obtained by these dosimeters is within 5% of the results obtained by FWT-60 radiochromic nylon dosimeters. This enables a user of low voltage EB equipment to evaluate its performance after routine maintenance within few hours versus few days using radiochromic nylon dosimeters.

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