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MEMORANDUM REPORT ARCCB-MR-92022

**DETERMINATION OF SANDOZ BLACK ALUMINUM
COLORING DYE, OLIVE ALUMINUM COLORING
DYE, AND SODIUM DICHROMATE ALUMINUM
SEALING SOLUTIONS BY UV-VISIBLE
SPECTROPHOTOMETRY**

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MAY 1992



**US ARMY ARMAMENT RESEARCH,
DEVELOPMENT AND ENGINEERING CENTER
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INTRODUCTION

The chemical literature lacks an acceptable method to determine and adequately control Sandoz black aluminum coloring dye, olive aluminum coloring dye, and sodium dichromate aluminum sealing solutions in aluminum finishing processes. Lack of optimization of these solutions causes serious problems for the aluminum finishing industry, such as poor quality products and wasted resources.

The only published chemical analysis methods to determine these solutions are the color comparison kits provided by Sandoz Corporation (refs 1-3). Lack of other chemical analysis methods may be because Sandoz solution chemistries are proprietary. Precisions by this method are poor due to only a relative-type measurement.

The specific method given here does provide acceptable analysis and control of these process solutions. The method consists of ultraviolet (UV)-visible spectrophotometry (refs 4-6).

EXPERIMENTAL PROCEDURE

Strict analytical chemistry methods and procedures are followed throughout this section. An excellent source of reference for these methods and procedures is by Fritz and Schenk (ref 4).

There are three separate chemical analysis methods presented here for Sandoz black aluminum coloring dye, Sandoz olive aluminum coloring dye, and Sandoz sodium dichromate aluminum sealants.

For the first dye, one analytical reagent grade standard solution is required. It is a 12.5 ± 0.1 -g/l Sandoz black dye solution that meets Sandoz Corporation specifications (ref 1). Three standards are prepared in 100-ml

volumetric flasks for UV-visible spectrophotometric analysis. The Sandoz black dye concentrations of these are 25.0, 12.5, and 0 parts per million (ppm). All Sandoz black dye solution samples are prepared in triplicate in 100-ml volumetric flasks for the dye analysis, and a 1:500 dilution is required of these sample solutions. The analytical system used is the Hewlett-Packard UV-visible spectrophotometer (Hewlett-Packard Corp., Palo Alto, CA). Hewlett-Packard publishes a manual that is an excellent source of reference for operation and maintenance of this instrument (ref 7). The operating conditions for the spectrophotometer are a 600-nm wavelength and 1-second integration time.

For the second dye, one analytical reagent grade standard solution is required. It is a 4.5 ± 0.05 -g/l Sandoz olive dye solution that meets Sandoz Corporation specifications (ref 2). Three standards are prepared in 100-ml volumetric flasks for UV-visible spectrophotometric analysis. The Sandoz olive dye concentrations of these are 9.0, 4.5, and 0 ppm. All Sandoz olive dye solution samples are prepared in triplicate in 100-ml volumetric flasks for the dye analysis, and a 1:500 dilution is required of these sample solutions. The analytical system used is the Hewlett-Packard UV-visible spectrophotometer. The operating conditions for the spectrophotometer are a 375-nm wavelength and 1-second integration time.

For the sealant, one analytical reagent grade standard solution is required. It is a 50.0 ± 0.5 -g/l Sandoz sodium dichromate sealant solution that meets Sandoz Corporation specifications (ref 3). Three standards are prepared in 100-ml volumetric flasks for UV-visible spectrophotometric analysis. The Sandoz sodium dichromate sealant concentrations of these are 50.0, 25.0, and 0 ppm. All Sandoz sodium dichromate sealant solution samples are prepared in triplicate in 100-ml volumetric flasks for the sealant analysis, and a 1:1000

dilution is required of these sample solutions. The analytical system used is the Hewlett-Packard UV-visible spectrophotometer. The operating conditions for the spectrophotometer are a 350-nm wavelength and 1-second integration time.

Using the procedures in this instrument's operating manual, standard and sample solution absorbance data for each method are recorded. Since the standard solution concentrations are known, sample solution concentrations can be calculated.

RESULTS AND DISCUSSION

The calibration data are given for the standard Sandoz black dye, olive dye, and sodium dichromate sealant solutions in Tables 1A, 1B, and 1C and are linear for the range given. If these data are found to be non-linear, then they must be acquired again.

Tables 2A, 2B, and 2C present the corresponding sample data for these respective solutions. These sample solutions are diluted 1:500 for dyes and 1:1000 for sealants to attain detector linearity and to minimize interferences.

Due to a linear operating range, the following simplified calculation is used to determine Sandoz black dye concentration in the original sample solutions:

$$\text{g/l dye} = (12.5)(\text{sample absorbance}/25.0 \text{ ppm standard absorbance}) \quad (1)$$

The Sandoz black dye solutions in Table 2A have a 10.4 and 12.2-g/l dye concentration for sample solutions one and two, respectively.

Table 3A shows that Sandoz black dye can be determined in solutions at the optimized wavelength of 600 nm using a 1:500 dilution.

Again due to a linear operating range, the following simplified calculation is used to determine Sandoz olive dye concentration in the original sample solutions:

$$\text{g/l dye} = (4.5)(\text{sample absorbance}/9.0 \text{ ppm standard absorbance}) \quad (2)$$

The Sandoz olive dye solutions in Table 2B have a 3.57 and 4.17-g/l dye concentration for sample solutions one and two, respectively.

Table 3B shows that Sandoz olive dye can be determined in solutions at the optimized wavelength of 375 nm using a 1:500 dilution.

Also due to a linear operating range, the following simplified calculation is used to determine Sandoz sodium dichromate sealant concentration in the original sample solutions:

$$\text{g/l dye} = (50.0)(\text{sample absorbance}/50.0 \text{ ppm standard absorbance}) \quad (3)$$

The Sandoz sodium dichromate sealant solutions in Table 2C have a 42.1 and 47.9-g/l sealant concentration for sample solutions one and two, respectively.

Table 3C shows that Sandoz sodium dichromate sealant can be determined in solutions at the optimized wavelength of 350 nm using a 1:1000 dilution.

It is useful to evaluate the variations in precision for the materials and methods used. Tables 4 through 8 present these data for the 100-ml class-A volumetric flask, 0.250-ml micropipet, 12.5-g/l Sandoz black dye standard solution, 4.5-g/l Sandoz olive dye standard solution, and 50.0-g/l Sandoz sodium dichromate sealant standard solution.

Variations in precision are also evaluated for the UV-visible spectrophotometer. Tables 9 through 11 present data for six consecutive replicates of the 25.0-ppm Sandoz black dye standard solution, 9.0-ppm Sandoz olive dye standard solution, and 50.0-ppm Sandoz sodium dichromate sealant standard solution, respectively.

These data by the given methods are sufficient to adequately control the dye and sealant concentrations according to standard operating procedures, thus providing efficient use of resources.

The optimum operating range of the Sandoz black aluminum coloring dye solution is 12.5 g/l maximum with resulting precisions in the 0.5 to 0.9-g/l range. The optimum operating range of the Sandoz olive aluminum coloring dye solution is 4.5 g/l maximum with resulting precisions in the 0.2 to 0.4-g/l range. The optimum operating range of the Sandoz sodium dichromate aluminum sealing solution is 50.0 g/l maximum with resulting precisions in the 1.5 to 2.0-g/l range. These methods provided adequate control of the three processes supported by six years of testing.

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1. "Sandoz Black Dye Color Comparison Procedure and Specifications," Sandoz Corporation, Hackensack, NJ, 1980.
2. "Sandoz Olive Dye Color Comparison Procedure and Specifications," Sandoz Corporation, Hackensack, NJ, 1980.
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7. "HP Diode-Array UV-Visible Spectrophotometer Operators Manual," Hewlett-Packard Corporation, Palo Alto, CA, 1988.

TABLE 1A. STANDARD SOLUTION DATA FOR SANDOZ BLACK DYE*

Replicate	Absor. (AU) 0 ppm Dye	Absor. (AU) 12.5 ppm Dye	Absor. (AU) 25.0 ppm Dye
1	0.000	0.175	0.360
2	0.000	0.175	0.350
3	0.000	0.175	0.355
X(avg)	0.000	0.175	0.355

*Each solution is a 500 dilution of a normal Sandoz black dye standard solution.

TABLE 1B. STANDARD SOLUTION DATA FOR SANDOZ OLIVE DYE*

Replicate	Absor. (AU) 0 ppm Dye	Absor. (AU) 4.5 ppm Dye	Absor. (AU) 9.0 ppm Dye
1	0.000	0.080	0.165
2	0.000	0.075	0.155
3	0.000	0.085	0.160
X(avg)	0.000	0.080	0.160

*Each solution is a 500 dilution of a normal Sandoz olive dye standard solution.

TABLE 1C. STANDARD SOLUTION DATA FOR SANDOZ SODIUM DICHROMATE SEALANT*

Replicate	Absor. (AU) for 0 ppm Sealant	Absor. (AU) for 25.0 ppm Sealant	Absor. (AU) for 50.0 ppm Sealant
1	0.000	0.250	0.505
2	0.000	0.250	0.495
3	0.000	0.255	0.500
X(avg)	0.000	0.252	0.500

*Each solution is a 1000 dilution of a normal Sandoz sodium dichromate sealant standard solution.

TABLE 2A. EXPERIMENTAL SAMPLE DATA FOR SANDOZ BLACK DYE SOLUTIONS*

Replicate	Dye Sample One Absor. (AU)	Dye Sample One Conc. (ppm)
1	0.295	20.8
2	0.295	20.8
3	0.295	20.8
X(avg)	0.295	20.8
Replicate	Dye Sample Two Absor. (AU)	Dye Sample Two Conc. (ppm)
1	0.340	24.0
2	0.345	24.4
3	0.345	24.4
X(avg)	0.343	24.3

*Each replicate is a 1:500 dilution of the original sample solution.

TABLE 2B. EXPERIMENTAL SAMPLE DATA FOR SANDOZ OLIVE DYE SOLUTIONS*

Replicate	Dye Sample One Absor. (AU)	Dye Sample One Conc. (ppm)
1	0.125	7.03
2	0.125	7.03
3	0.130	7.31
X(avg)	0.127	7.14
Replicate	Dye Sample Two Absor. (AU)	Dye Sample Two Conc. (ppm)
1	0.150	8.44
2	0.150	8.44
3	0.145	8.16
X(avg)	0.148	8.33

*Each replicate is a 1:500 dilution of the original sample solution.

**TABLE 2C. EXPERIMENTAL SAMPLE DATA FOR SANDOZ
SODIUM DICHROMATE SEALANT SOLUTIONS***

Replicate	Sealant Sample One Absor. (AU)	Sealant Sample One Conc. (ppm)
1	0.425	42.4
2	0.420	41.9
3	0.420	41.9
X(avg)	0.422	42.1
Replicate	Sealant Sample Two Absor. (AU)	Sealant Sample Two Conc. (ppm)
1	0.480	47.9
2	0.480	47.9
3	0.480	47.9
X(avg)	0.480	47.9

*Each replicate is a 1:1000 dilution of the original sample solution.

TABLE 3A. WAVELENGTH OPTIMIZATION FOR SANDOZ BLACK DYE SOLUTIONS*

Wavelength (nm)	Sandoz Black Dye 25.0 ppm Absor. (AU)
325	0.245
350	0.235
375	0.210
400	0.195
425	0.200
450	0.190
475	0.185
500	0.200
525	0.235
550	0.280
575	0.325
600	0.355
625	0.340

*The concentration is a 1:500 dilution of a normal Sandoz black dye solution.

TABLE 3B. WAVELENGTH OPTIMIZATION FOR SANDOZ OLIVE DYE SOLUTIONS*

Wavelength (nm)	Sandoz Olive Dye 9.0 ppm Absor. (AU)
325	0.135
350	0.155
375	0.160
400	0.130
425	0.095
450	0.075
475	0.060
500	0.060
525	0.075
550	0.090
575	0.105
600	0.100
625	0.085

*The concentration is a 1:500 dilution of a normal Sandoz olive dye solution.

TABLE 3C. WAVELENGTH OPTIMIZATION FOR SANDOZ SODIUM DICHROMATE SEALANT SOLUTIONS*

Wavelength (nm)	Sandoz Sealant 50.0 ppm Absor. (AU)
325	0.375
350	0.500
375	0.340
400	0.140
425	0.085
450	0.070
475	0.040
500	0.015
525	0.005
550-625	0.000

*The concentration is a 1:1000 dilution of a normal Sandoz sodium dichromate sealant solution.

TABLE 4. PRECISION OF THE 100-ml CLASS-A VOLUMETRIC FLASK

Replicate	Volume (ml)*
1	100.14
2	99.97
3	99.89
4	100.12
5	100.03
6	100.06
X(avg)	100.04
Sn	0.09

*Volumes are calculated from the weight-volume relationship of the contained deionized water solution corrected for temperature.

TABLE 5. PRECISION OF THE 0.250-ml MICROPIPET

Replicate	Volume (ml)*
1	0.2582
2	0.2497
3	0.2546
4	0.2545
5	0.2557
6	0.2532
X(avg)	0.2543
Sn	0.0028

*Volumes are calculated from the weight-volume relationship of a pipetted deionized water solution corrected for temperature.

TABLE 6. PRECISION OF THE 12.5-g/l SANDOZ BLACK DYE STANDARD SOLUTION

Replicate	Sandoz Black Dye Conc. (g/l)*
1	12.5
2	12.3
3	12.7
4	12.4
5	12.8
6	12.3
X(avg)	12.5
Sn	0.21

*Sandoz black dye concentrations are determined using UV-visible spectrophotometry.

TABLE 7. PRECISION OF THE 4.5-g/l SANDOZ OLIVE DYE STANDARD SOLUTION

Replicate	Sandoz Olive Dye Conc. (g/l)*
1	4.55
2	4.45
3	4.40
4	4.60
5	4.50
6	4.50
X(avg)	4.50
Sn	0.07

*Sandoz olive dye concentrations are determined using UV-visible spectrophotometry.

**TABLE 8. PRECISION OF THE 50.0-g/l SANDOZ SODIUM
DICHROMATE SEALANT SOLUTION**

Replicate	Sandoz Sealant Conc. (g/l)*
1	49.0
2	49.5
3	50.5
4	50.5
5	50.0
6	50.0
X(avg)	49.9
Sn	0.6

*Sandoz sodium dichromate sealant concentrations are determined using UV-visible spectrophotometry.

**TABLE 9. PRECISION OF THE 25.0-ppm SANDOZ BLACK DYE
SOLUTION BY UV-VISIBLE SPECTROPHOTOMETRY**

Replicate	Absor. (AU) 25.00 ppm Dye
1	0.360
2	0.350
3	0.355
4	0.360
5	0.350
6	0.355
X(avg)	0.355
Sn	0.004

TABLE 10. PRECISION OF THE 9.0-ppm SANDOZ OLIVE DYE SOLUTION BY UV-VISIBLE SPECTROPHOTOMETRY

Replicate	Absor. (AU) 9.0 ppm Dye
1	0.165
2	0.155
3	0.160
4	0.155
5	0.165
6	0.160
X(avg)	0.160
Sn	0.004

TABLE 11. PRECISION OF THE 50.0-ppm SANDOZ SODIUM DICHROMATE SEALANT SOLUTION BY UV-VISIBLE SPECTROPHOTOMETRY

Replicate	Absor. (AU) 50.0 ppm Sealant
1	0.505
2	0.495
3	0.500
4	0.505
5	0.490
6	0.505
X(avg)	0.500
Sn	0.006

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