

Evaluation of the image quality of a radiographic film emulsion AGFA D7 exposed by X-rays and developed by machine processing 12200 Berlin, Germany (8 min cycle time at 29 °C) using Dürr NDT XR D-6 NDT developer P: +49 30 8104-0 and XR F-6 NDT fixer (new formulas)

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**BAM** reference

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Customer

DÜRR NDT GmbH & CO. KG

Höpfigheimer Straße 22 74321 Bietigheim-Bissingen

Order date

2016-03-21

Reference

Your order No. N1100663 from 2016-03-30

**Test samples** 

Dürr NDT XR D-6 NDT developer (new formula, HFE-DEV 05022016, exp. date 2018-02) and XR F-6 NDT fixer (new formula, HFE-FIX 21012016, exp. date 2018-02) and film batch AGFA D7, emulsion number 527 0036 (2018-07) in format 10x24 cm<sup>2</sup>, vacupacked with 27 µm

lead screens on both sides)

Receipt of samples

2016-03-10

**Test date** 

March to May 2016

**Test location** 

**BAM Berlin** 

Test procedure

according to

DIN EN ISO 11699-1:2012 and ASTM E1815-08, film development in Dürr NDT XR D-6 NDT developer

and XR F-6 NDT fixer (new formulas), automatic processing in AGFA NDT S machine with 8 min cycle

time, immersion time 100s, temperature 28 °C

Report date

2016-05-30

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The test results refer exclusively to the tested materials.

### **Scope of the Test:**

Determination of the sensitometric properties, film system class and the measurement of the ISO speed of a non-destructive testing film batch of AGFA D7 when exposed to X-ray radiation and automatically processed in the chemistry Dürr NDT XR D-6 NDT developer and XR F-6 NDT fixer (new formulas), automatic processing in AGFA NDT S machine (type 5320/100), operating with 8 min cycle time, an immersion time of 100s and a developer temperature of 29 °C, according to DIN EN ISO 11699-1:2012 and ASTM E1815-08.

## **Test Program:**

The measurement of sensitometric properties of the exposed and processed films, the classification of the evaluated film system and the measurement of the ISO speed was carried out according to the DIN EN ISO 11699-1:2012 and ASTM E1815-08 as follows:

- 1. Exposure of the films with X-ray radiation in dose steps using an automatic film transport unit according to the standards of the test procedure.
- Processing of the films according to the above customer defined parameters in an automatically controlled AGFA NDT S film processor.
- 3. Measurement of the diffuse optical densities of the exposed and processed films to determine the sensitometric properties of the film and to calculate the film gradients at  $D-D_0 = 2.00$  and  $D-D_0 = 4.00$ .
- 4. Measurement of the granularity at D-D<sub>0</sub> =  $2.00 \pm 0.05$ .
- Control of the processing chemistry in the AGFA NDT S processor on basis of DIN EN ISO 11699-2:2012 using certified pre-exposed AGFA PMC film strips.

#### Instruments for processing and measurements:

The exposures of the films (10x24 cm, vacupacked with 0.027 mm front and back lead screens) were performed with the filtered (8 mm Cu) 220 kV radiation of a highly stabilized X-ray facility (320 Isovolt D for dosimetry of the Seifert company) and an automated computer controlled film transport system.

A calibrated Macbeth TD 502 densitometer was used for the measurement of the diffuse optical density (2 mm aperture diameter). The granularity was measured with a computer controlled microdensitometer of the type Joyce/Loebl 3CS using a circular aperture of 100  $\mu$ m diameter.

#### Film processing:

All films were automatically processed using an AGFA NDT S film processor, Dürr NDT XR D-6 NDT developer (new formula, HFE-DEV 05022016, exp. date 2018-02) and XR F-6 NDT fixer (new formula, HFE-FIX 21012016, exp. date 2018-02) at a developer temperature of 29 °C, with an immersion time of 100 s and a process cycle time of 8 minutes.

# Procedure for determination of the film system parameters according to DIN EN ISO 11699-1:2012 and ASTM E1815 – 08 (reapproved 2013):

The system parameters gradient G(2) and G(4) at the optical densities 2.00 and 4.00 above fog and base  $(D_0)$ , the granularity  $\sigma_D$  and the gradient/noise ratio  $G(2)/\sigma_D$  are determined for the film system, which consists of the combination of the selected film type and film processing carried out at the above described specified processing parameters.

In detail the following steps were carried out:

- procurement of films processing chemicals and test strips,
- preparation of the developer and fixer according to the prescriptions of the manufacturer,
- test of the specified composition chemistry with pre-exposed film test strips at the specified immersion time and developer temperature,
- stabilisation of the developer with approximately 0.6 m<sup>2</sup> film material,
- test exposure experiments for the determination of the suitable exposure times needed for the steps of the desired optical density,
- exposure of 8 film samples for each system with filtered 220 kV radiation on at least twelve steps to cover the required optical densities between 1.0 and 4.5 above fog ( $D_0$ ) according to the procedure in DIN EN ISO 11699-1:2012 and ASTM E1815-08.
- processing of the films according to the above described parameters,
- daily control of the film processing system by film strips according to DIN EN ISO
   11699-2:2012 before and after the development of the exposed films,
- measurement of the diffuse optical densities of the processed films with the Macbeth densitometer and subsequent recordings of the exposure dose, the corresponding measured density values, determined from an average of three values measured at each uniformly exposed density step area, as well as the optical density D<sub>0</sub> of the fog and base at an unexposed film area,
- approximation of the D versus K curve with densities (D-D<sub>0</sub>) by a third order polynomial and calculation of the gradient G according to equation (1):

$$G = \frac{K}{\log_{10} e} \cdot \frac{dD}{dK} \tag{1}$$

K - exposed dose, required for density  $(D-D_0)$ ;

- determination of the gradient G(2) for the density D -  $D_0$  = 2.00 and the gradient G(4) at D -  $D_0$  = 4.00 in accordance with DIN EN ISO 11699-1:2012 and ASTM E1815-08 from the G versus K curve.

- measurement of the local density fluctuations of the films at density D-D<sub>0</sub> =  $2.00 \pm 0.05$  with a microdensitometer (scan step 100  $\mu$ m, scan length 140 mm),
- calibration of the microdensitometer into diffuse optical density units with the aid of a film sample of the investigated film system,
- calculation of the granularity  $\sigma_D$  from the root of the variance of the optical density values after conversion of the specular densities into diffuse densities, a digital high pass filtering to cut off low spatial frequency noise below 0.1 mm<sup>-1</sup> and the determination of the median according to ISO 10505:2009 and DIN EN ISO 11699-1:2012,
- conversion of the granularity value into the corresponding value for measurements with a circular aperture with a diameter of  $100 \mu m$ ,
- correction of the granularity value on the basis of measured density above fog and base to  $D-D_0 = 2.00$ ,
- calculation of the average of the relevant data from at least 6 film samples.

#### Results:

The diffuse density of fog and film base results in:

$$D_0 = 0.21 \pm 0.01$$

The characteristic curve of the films is shown in figure 1 up to the density 4.5 above fog and base. This  $(D-D_0)$  versus K plot enables the estimation of the exposed dose  $K_s$  for density 2 above fog and base to:

$$K_S = (3.3 \pm 0.1) \text{ mGy}$$

This corresponds according to Table 2 of DIN EN ISO 11699-1:2012 to an ISO speed S of:

$$S = 320$$

The values of the gradients determined under the described conditions amount to:

$$G(2) = 4.39 \pm 0.03$$

$$G(4) = 7.57 \pm 0.09$$

The granularity  $\sigma_D$  at density D - D<sub>0</sub> = 2.00 results in:

$$\sigma_D$$
 = 0.0312 ± 0.0011

The gradient/noise ratio is calculated to:

$$G(2) / \sigma_D = 141 \pm 6$$

Figure 1 shows the mean  $(D-D_0)$  densities of eight films and the mean of the according calculated gradients as function of the exposure dose K.

# Measurement uncertainty and decision rules:

Measurement uncertainty is determined according to GUM<sup>1</sup>. The presented mean values and their uncertainties were determined from the respective values of the gradients and the granularity at the given densities (D - D<sub>0</sub>) = 2.00 and (D - D<sub>0</sub>) = 4.00 from 6 film samples (trimmed mean). The uncertainties of the gradients  $U_{G(2)}$ ,  $U_{G(4)}$ , the granularity  $U_{\sigma}$  and the gradient/noise ratio  $U_{G/\sigma}$  are obtained by multiplying the appropriate standard uncertainties by the coverage factor k= 2.57 based on the t-distribution for five degrees of freedom. The k factor corresponds for a t-distribution in case of a two tail region to a coverage probability of 95 % and of a one tail region to 97.5 % respectively.

The limiting values for the Film system class **C5** amount according to Table 1 of DIN EN ISO 11699-1:2012 to:

$$G_{min}$$
 (2) = 3.8,  $G_{min}$  (4) = 6.4,  $\sigma_{D,max}$  = 0.032,  $(G/\sigma_{D})_{min}$  = 120

The limiting values for the ASTM system Class **II** are defined according to Table 1 of ASTM E1815-08 as follows:

$$G_{min}$$
 (2) = 3.8,  $G_{min}$  (4) = 6.4,  $\sigma_{D,max}$  = 0.032,  $(G/\sigma_{D})_{min}$  = 120

The following decision rule for providing conformance with a confidence level of 97.5 % with the specified limits of film system classification according to the standards DIN EN ISO 11699-1:2012 and ASTM E1815-08 was applied.

$$[G~(2)-U_{G(2)}] \geq G_{min}~(2),~[G~(4)-U_{G(4)}] \geq G_{min}~(4),~[\sigma_D+U_\sigma] \leq \sigma_{D,max},~[G~(2)/\sigma_D-U_{G/\sigma}] \geq (G/\sigma_D)_{min}$$

The value [G (2) -  $U_{G(2)}$ ] may fall short by maximum 5 % of the limit of Table 1 of DIN EN ISO 11699-1:2012 and ASTM E1815-08 and the value of [G (4) -  $U_{G(4)}$ ] may fall short by 7 % of the limit of Table 1 of DIN EN ISO 11699-1:2012 and ASTM E1815-08 as well as the value of [ $\sigma_D$  +  $U_\sigma$ ] may exceed the limit of Table 1 of DIN EN ISO 11699-1:2012 and ASTM E1815-08 by 10 %, if the value [(G (2) /  $\sigma_D$ ) -  $U_{G/\sigma}$ ] does not exceed the limit value of Table 1 of DIN EN ISO 11699-1:2012 and ASTM E1815-08.

<sup>&</sup>lt;sup>1</sup> ISO/IEC Guide 98-3:2008-09 Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)

#### Film system evaluation:

The mixed film system consisting of the industrial radiographic film AGFA D7 (emulsion number 527 0036 (2018-07)), and the processing Dürr NDT XR D-6 NDT developer (new formula, HFE-DEV 05022016, exp. date 2018-02) and XR F-6 NDT fixer (new formula, HFE-FIX 21012016, exp. date 2018-02) comply with the limiting value specifications of DIN EN ISO 11699-1:2012 and ASTM E 1815-08 according to the above decision rules. Following, the above tested film system meets the requirements of the Film system class **C5** of the standard DIN EN ISO 11699-1:2012 and ASTM System **Class II** of the standard ASTM E1815-08 in case of automatic processing at a cycle time of 8 min, at an immersion time of 100 s, and at a developer temperature of 29 °C.

## Thio-Test according to ISO 18901:2010:

The achieved processing quality was evaluated by applying the AGFA Thio-Test on the processed films. The test correlates with international standards (ISO/ANSI) to determine the archival quality of industrial X-ray films. The colour comparison of test spot with the light step indicate according to ISO 18901:2010 a Life Expectancy of L.E. = 500.

The result means a Life Expectancy of 500 Years for the developed films.

#### Quality assurance of the processing chemistry:

Pre-exposed and certified AGFA PMC film test strips (Batch C 147, Expiration Date 2016-07) were used to check the specified developer system.

The strips exhibit a diffuse density of fog and film base  $D_0 = 0.26 \pm 0.01$ .

Following, the used test strips have a reference speed index  $S_r$  = 2.01 (step 3) and a reference contrast index  $C_r$  = 1.32 (step 7 – step 3) according to DIN EN ISO 11699-2:2012, but using AGFA G135 developer at 100s and 29 °C. The film test strips showed the following values from beginning until end of the test period:

$$S_x = 1.91 \pm 0.02$$
 (-5 % relative to  $S_r$ ) and  $C_x = 1.34 \pm 0.02$  (+2 % relative to  $C_r$ )

This indicates that the requested conditions (100 s immersion time, 29 °C and Dürr NDT chemistry with new formula) are within the AGFA specification for their films in their chemistry. The maximum deviation from the reference values according to ISO 11699-2 is  $\pm$  10 %. If this cannot be achieved for the standard conditions (8 min and 29 °C), than the developer temperature can be adapted by  $\pm$  2 K.

Therefore, the quality assurance of the mixed system of an AGFA D5 film and Dürr NDT XR D-6 NDT developer and XR F-6 NDT fixer (new formulas) can be performed by using the reference values of the certified PMC strips provided by AGFA for their chemistry G 135.

This is an important simplification of quality assurance for the user of such a mixed system and follows the description of quality assurance for mixed systems according to the D2 guideline for dark room processing of DGZfP (German NDT society).

#### Remark:

This report summarizes only the results of the measurements at a single emulsion and is valid only for this special emulsion; especially it states no general classification of a film system. It is valid only for the specially selected mixed film system. For a system classification corresponding to DIN EN ISO 11699-1:2012 a continuous measurement of all films of all manufactured emulsions by an independent third party is necessary, or, the continuous own product control in the house of the manufacturer. Additionally, the participation in a proficiency test according to DIN EN ISO 11699-1:2012 is required for the harmonisation of the measuring procedures and algorithms as well as sample test measurements by an independent third party.

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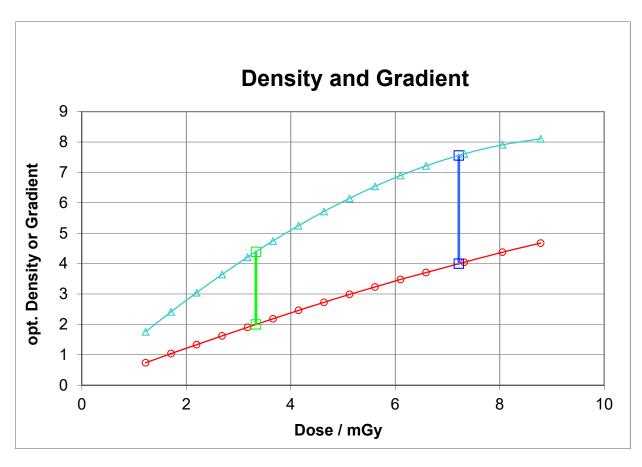
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**Figure 1:** Mean optical density <D- $D_0>$  (red curve) and mean gradient <G> (blue curve) of eight films as function of exposure dose K.