METALS

Deconstructing Incralac: A formulation study of acrylic coatings for the protection of outdoor bronze sculpture

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ABSTRACT

Incralac is a clear coating designed as a lacquer for copper alloys in the 1960s by the International Copper Research Association and the British Non-Ferrous Metals Research Association. Incralac was adopted by conservators for the protection of outdoor bronzes and remains a popular choice in spite of mixed reviews on its performance. Over time, manufacturers of Incralac have employed different additives and solvents: a water-based version has also been produced. The uncertainties associated with using proprietary products prompted a study designed to compare currently available commercial Incralac with laboratory-made replacements based on the earliest published formulation but using less toxic solvents. The study also includes the commercial lacquer Permalac. Brass coupons were coated with nine different coatings and naturally aged. Results show a similar performance between Incralac, Permalac, and a laboratory-made imitation Incralac.

INTRODUCTION

The protective clear coating called Incralac was developed in the 1960s based on research initiated jointly by the International Copper Research Association (INCRA) – now the International Copper Association – and a cohort from the British Non-Ferrous Metals Research Association (BNFMRA). Both institutions determined that Acryloid B-44 manufactured by Rohm & Haas Co. – now Paraloid B-44 by Dow Chemical – gave superior protection for an air-drying coating on copper alloys and had good appearance and working properties (Bharucha 1965, INCRA 1966). Paraloid B-44 (hereafter B-44) is a methyl methacrylate copolymer with minor copolymer units of ethyl acrylate and butyl methyl acrylate (Chiantore 1996, Brostoff 2003, Rohm and Haas 2007). INCRA and BNFMRA developed formulations that contained 30 wt% B-44 in toluene and butyl acetate (or ethyl alcohol), benzotriazole (BTA) as a corrosion inhibitor, and either silicone fluid or epoxidized soybean oil (ESBO) as a leveling agent.

Art conservators quickly adopted the coating for the protection of outdoor bronze sculpture and it remains a popular choice today. A substantial amount of research has been carried out on Incralac over the past 10–15 years through its inclusion in a number of ageing and weathering studies. The performance can vary depending on chemical patinas (Brostoff 2003), solvent carriers, and additives. The coating was trademarked under the name Incralac, but it was never patented and manufacturers have been free to modify the formulation at will. In fact, environmental regulations have prompted changes in solvent in order to reduce the volatile organic compounds (VOCs) and the solvent-based coating will likely be replaced with a new water-based product line.

A review of the development of Incralac and modifications made over the past few decades was the springboard to designing an experiment on lab-made versions. Testing different formulations in the laboratory could provide another avenue for conservators and scientists to ascertain the effects of formulation changes. Preparing Incralac coatings in the laboratory could also provide more control in treatments and coating studies, and perhaps help to improve its performance on outdoor sculpture. Furthermore, exploring different solvents could lead to less toxic formulations that reduce health risks. This paper presents a study that compared lab-made Incralac formulations with commercial Incralac (water and solvent-based)

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ABOUT THE STUDY

Coatings tested

Nine acrylic coatings were compared in this study including five lab-made imitations of Incralac, solvent and water-based Incralac, and Permalac (Table 1). Coatings #1–5 were prepared in the lab using B-44 and 1-methoxy-2-propanol – a low-toxicity solvent having a similar evaporation rate to xylene (both 0.75) (PPG 2008, Shell Chemicals 2011). 1-methoxy-2-propanol has also been suggested as an alternative to toluene and xylene

Table 1. Formulation of the coatings as-sprayed on the test coupons given in weightpercentage. The proportions and components for the proprietary products are extrapolatedfrom the product safety and technical data sheets provided by manufacture (StanChem 2006,Peacock Laboratories 2007, StanChem 2015). The formulation developed by BNFMRA is listedfor reference (INCRA 1966)

Components	BNFMRA formulation 1963 ⁺	Imitation Incralac	Imitation Incralac no BTA	Imitation Incralac BYK	Imitation Incralac G-62	Imitation Incralac A-8000	Incralac matte∧	Incralac∧	Incralac, water-based	Permalac satin*
Coating #		1	2	3	4	5	6	7	8	9
Paraloid B-44	8	8	8	8	8	8	8	8		
Paraloid B-48N [‡]										10
Acrylic urethane									Х	
Benzotriazole	0.12	0.11			0.11	0.11	0.3	0.3		
Paraplex G-60	0.12	0.78	0.78				0.3	0.3		
Paraplex G-62					0.78					
Paraplex A-8000						0.78				
BYK-4513				0.39						
Ceramat 258				0.90						
StanChem flattening agent							1.0			
UV absorber							0.2	0.2		
1-methoxy-2-propanol		91	91	91	91	91				
Toluene	90						63	64		85
Xylene							27	27		
Butanone							1	1		
N-Butyl acetate										5
N-Methylpyrrolidone									2–3	
Triethylamine									1–2	
Ethyl alcohol	1.3									
Methyl cellosolve	0.6									
Approximate wt% resin solids	8	8	8	8	8	8	8	8		10

⁺ The formulation developed by BNFMRA was published as a 30 wt% solids solution in 1963 and intended for dilution with toluene for spraying; therefore, the formula was converted for this table to a comparable ratio to the coatings in this study (INCRA 1966).

^A The Incralac coatings (# 6 and 7) have a 15 wt% solids concentrate from the manufacturer with a solvent ratio of 54:2:28 with toluene:butanone:xylene; toluene was used to dilute the concentrate for spray application. No analysis was done to confirm, but the ESBO is assumed to be G-60.

^{*} The Permalac (#9) was not diluted and sprayed as manufactured (10 wt% solids).

* Based on FTIR analysis.

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Figure 1. FTIR spectrum overlay of Permalac (red), Paraloid B-48N (blue), Incralac (purple) and Paraloid B-44 (green)

for other Paraloid resins (Phenix 1993, Rohm and Haas 2006). Coating #1 is the most direct copy of the 1963 BNFMRA formulation and includes BTA and Paraplex G-60 (ESBO). Coating #2 was prepared without BTA to provide a control for its impact as a corrosion inhibitor, as shown by previous studies (INCRA 1966, Brostoff 2003, Allam 2009, CDA 2009).

Coating #3 was formulated using alternate additives manufactured by BYK-Chemie (hereafter BYK). This formulation incorporated BYK-4513, a proprietary polymer designed to promote adhesion to metal substrates and provide corrosion resistance (BYK 2014), as well as Ceramat 258, an oxidized high-density polyethylene wax dispersion designed to reduce gloss and improve scratch resistance (BYK 2013). Other plasticizers manufactured by Hallstar were used in coatings #4 and 5. The Paraplex G-62 (hereafter G-62) used in coating #4 was suggested by technical support personnel at Hallstar for its greater stabilizing properties compared to Paraplex G-60 (found in coating #1). Both are high-molecular-weight soybean oil epoxides with good heat and light stability. Coating #5 contains the plasticizer Paraplex A-8000 (hereafter A-8000), which is a low-molecular-weight polyester adipate that could provide better permanence, volatility, and migration resistance than monomeric plasticizers. The lack of volatile organic compounds in this product should improve flexibility of the coating.

Coatings #6, 7, and 8 are Incralac products applied to specification. Mixtures #6 and 7 were diluted with toluene to improve spraying performance. The high gloss of Incralac can be a negative aesthetic characteristic and is often matted down by conservators; therefore, coating #6 contains the proprietary Incralac matting agent based on polyethylene (StanChem 2015). Coating #8 is the water-based Incralac designed by StanChem to lower VOCs. It should be noted that it has a very different formulation based on an acrylic-urethane resin.

Permalac is another proprietary product developed in 1995 that is an air-drying lacquer manufactured by Peacock Laboratories. The primary difference between Permalac and Incralac appears to be that the former replaces B-44 with B-48N resin. This observation is based on FTIR analysis of Permalac films which show increased intensity of methylene C-H stretching vibrations at 2950 cm⁻¹, due to the presence of butyl functional groups very similar to Paraloid B-48N resin spectra (Figure 1). B-48N was developed several years after B-44 specifically for better adhesion to metal. At the time of this study, Permalac was available for purchase as satin or matte, and the satin product was chosen for coating #9.

Sample preparation

A set of six metal coupons were prepared for each coating in the study: three for natural ageing and three to be used as controls. Coupons were made using brass sheet (90 Cu wt%, 10 Zn wt%) sheared into 7-cm squares. One side was abraded with Scotch-Brite pads and they were washed with water and Orvus detergent, rinsed with deionized water, and dried with cotton pads. Prior to coating, the surface was wiped with petroleum ether on cotton pads and cleaned with carbon dioxide blasting. A duplicate set of coupons were made on glass microscope slides.

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Figure 2. Scans of the coupons grouped to show before ageing (left) and after ageing (right)



Figure 3. Detail of each aged coupon at 50× magnification

The metal coupons were sprayed twice using an HVLP spray gun with the goal of achieving a 25- μ m-thick film. Due to the hand spray application, there are variations in the resulting thicknesses (Table 2) and sometimes in surface quality. The coupons were left to dry for two weeks and scanned on a flatbed scanner with a Qpcard 201 card for color reference. The control samples were stored in a dark, corrosion-protective zip-lock bag on a vertical rack.

Ageing the coupons

Three metal coupons per coating were exposed to the natural environment according to ASTM G50-10 on the roof of the Getty Center in Los Angeles, California for eleven months. The urban environment has a dry, subtropical climate that is moderately warm year-round having a mild rainy season in the winter months. Coupons were wiped with a cotton pad damp with water and scanned every two weeks.

EVALUATION

Application and visual assessment

Permalac (#9) and matte Incralac (#6) were easy to apply and produced a satin, even surface quality. Incralac (#7) also sprayed well and appeared glossy. The imitation Incralac mixtures sprayed well (#1, 2, 3, and 4); however, the coating containing A-8000 (#5) was prone to reticulation. The imitation Incralac containing G-62 (#4) sprays notably better than the coating with G-60 (#1). The water-based Incralac (#8) was harder to spray and appears cloudy and plastic.

Within the set of three coupons per coating, visual inspection showed that all performed very similarly over the course of eleven months; Figure 2 shows one representative coupon before and after ageing. The metal substrate for all coupons oxidized slightly – not attributable to film yellowing as aged coatings on glass slides consistently did not yellow. The coatings that prevented oxidation most effectively include imitation Incralac #1, matte Incralac #7 and Permalac #9. Corrosion became visible after two weeks of outdoor exposure for imitation Incralac without BTA (#2), as expected. The other imitation Incralac coatings with mild change include pitting with BYK (#3) and Incralac (#7), and very light-orange spotting with G-62 (#4) (Figure 3). The water-based Incralac (#8) formed a very fine craquelure that caused pitting corrosion (also observed in Brostoff 2003). After two months on the roof and subject to heavy rain, the water-based Incralac (#8) became blanched; the matte Incralac (#6) showed the highest surface tension to droplets.

Coatings were measured for gloss using a three-geometry goniophotometer. Incralac (both #7 and 8) and imitation Incralac (#1, 2, 4, and 5) had the highest gloss units, while the coatings with matting agents (#3, 6, and 9) were expectedly lower (Table 2). Most of the naturally aged coupons did not show significant change in gloss over eleven months, with the exception of water-based Incralac (#8) which decreased by 68%. The increase in gloss for #5 speculatively may be due to changes due to the A-8000 over time.

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Figure 4. Results of adhesion testing on imitation Incralac without BTA (#2) before ageing (left) and after ageing (right)

Table 2. Coating properties before and after natural ageing

	Thickness (µm)*		Gloss @ 60°		Adhe	sion [‡]	Pencil ha	ardness	Scratch resistance		
#	Before	After	Before	After	Before	After	Before	After	Before	After	
1	12.0	10.2	64	64	4B	2B	Н	4H	F	4H	
2	10.0	9	70	70	0B	1B-2B	HB	6H	HB	4H	
3	11.2	8.8	33	32	4B	4B	5H	6H	3H	4H	
4	7.6	6.6	56	60	4B-5B	4B-5B	6H	6H	4H	4H	
5	12.2	11	60	70	3B	3B-4B	Н	HB	F	НВ	
6	11.6	10.4	28	29	2B-3B	2B	F	4H	F	4H	
7	8.6	6.8	70^	63^	4B-5B	4B	5H	5H	4H	4H	
8	10.6	9	65^	21^	5B	5B	Н	3H	HB	н	
9	7.2	6.6	59^	56^	4B	5B	4H	6H	3H	3H	

Thickness meter accuracy is \pm (2.0 μ m + 3%).

Hardness and scratch resistance ranked from hard to soft by 6H-5H-4H-3H-2H-H-F-HB-B.

* Ranking is done on a scale from no detachment (5B, good adhesion) to more than 65% detachment (1B, poor adhesion).

[^] An average of two coupons instead of three due to varied spray application for these coupon sets.

Physical properties

The adhesiveness of each coating was measured before and after ageing using a tape test in accordance with ASTM D3359-09e2, Method B (crosshatch) (Table 2). The difference between unaged imitation Incralac #1 and 2 demonstrates that benzotriazole improves coating adhesion, though the adhesiveness of coating #2 does improve when aged eleven months (Figure 4). Other imitation Incralac formulations with BYK (#3) and G-62 (#4) maintain good adhesion before and after ageing. Incralac (#7) and Permalac (#9) show increased adhesion after ageing. Water-based Incralac (#8) shows the best adhesion overall.

The hardness of each coating was measured before and after ageing using a Pencil Hardness Tester according to ASTM D3363 (Table 2). After natural ageing, most of the coatings hardened with the exception of imitation Incralac with A-8000 (#5) that became softer. Imitation Incralac with G-62 (#4) and Incralac (#7) stayed the same and were the hardest coatings with the most scratch resistance. Interestingly, the G-62 (#4) produced a harder film and greater scratch resistance than G-60 (#1). The addition of a matting agent to Incralac (#6) reduced its film hardness.

Solubility

Since removability of Incralac has been a controversial issue, the solubility of the coatings was evaluated by adapting a method developed by Feller and Bailie (1972) using solvent mixtures with increasing solvency and a consistent 1:1 ratio of dipolar and hydrogen-bonding forces. Seven solvent mixtures containing toluene and acetone were prepared having a range of values in dispersion forces (f_d) and each mixture was applied to the films by cotton swab (Figure 5).

Imitation Incralac without BTA (#2) retained its solubility in all mixtures before and after ageing, while the coating #1 with BTA showed decreased solubility. This suggests that the reactivity of BTA with copper may hinder film solubility. All other coatings decreased in solubility after ageing except for coating #8, the only water-based coating. This could be due to the propagation of cracks and pores in the film which allowed for solvent uptake. All aged

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Figure 5. Solubility of the coatings on control samples (C) and after natural ageing (N). Smaller f_d number indicates a stronger solvent power. * indicates the solvent mixture that most effectively removed the coating

Electrochemical impedance spectroscopy (EIS)

The impedance (Z) at low frequency (0.1 Hz) is a good indicator of performance, being equivalent to the total resistance of the coating (Loveday 2004). With the exception of water-based Incralac (#8), all coatings demonstrate very high resistance (>1 \times 10⁸ Ω) at low frequency, which indicates high performance and excellent barrier properties of the Paraloid resins (Figure 6). After ageing, the coatings show a decrease in resistance of up to 2 orders of magnitude. The coating with the largest decrease in performance was coating #2 (without BTA). While showing a high Z in the control, the coating does not stand up as well to ageing due to the lack of protective Cu-BTA chelates on the metal surface (Madsen 1967). The coating which showed the least decrease in resistance on ageing was the matte Incralac (#6). Speculatively, this could be due to crosslinking of the polyethylene-based matting agent, which in turn improves the coating barrier properties. The performance of the water-based Incralac (#8) is lower than all the other coatings. This could be due in part to poor film formation compared to coatings containing more rapidly evaporating solvents such as toluene and 1-methoxy-2-propanol or being prone to water absorption as seen by blanching after rain.



Figure 6. Performance of all coatings before and after ageing in outdoor conditions for eleven months. Coating performance is measured as the impedance (Z) at 0.1 Hz. Z is corrected for thickness (Table 2)

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Fourier transform infrared spectroscopy (FTIR)

Infrared analysis was performed on the Incralac (#7), Permalac (#9), and imitation Incralac (#1) sprayed on glass slides to observe the effects of outdoor weathering. Spectral comparisons between aged Incralac (#7) and imitation Incralac (#1) show a slight broadening in the carbonyl band at 1725 cm⁻¹, reduction in CH stretching at 2950 cm⁻¹ and minor increase in OH vibrations at 3400 cm⁻¹, particularly in #1. Comparison of aged Incralac and Permalac show overall spectral broadening of the carbonyl band, increases in the CO and OH modes located at 1300–1000 cm⁻¹ and additional minor increase in the OH vibration at 3400 cm⁻¹ in #7. Incralac (#6) containing a matting agent shows a slight decrease in methyl and methylene functionalities at 2849 and 2925 cm⁻¹, likely due to oxidative changes to the surface of the coatings. Infrared analysis showed overall chemical changes in all of the aged coatings. The degree of change might be not only due to the composition of the copolymers but also to the synergistic influences of various additives.

CONCLUSION

Nine coatings related to Incralac were evaluated by a multi-analytical approach to compare application, performance, appearance, and reversibility. The study showed that a lab-made low-toxicity Incralac using 1-methoxy-2 propanol can provide protection on brass coupons similar to the commercial product. The performance of Permalac was similar to that of Incralac, whereas the water-based Incralac – developed in conformance to environmental regulations – performs much worse. Deconstructing the formulation of Incralac by designing a series of imitation coatings having different additives revealed some trends, but additionally exposed new research areas that can be explored in the future; for example, comparing chemical change using gas chromatography/mass spectrometry (GC/MS), performance on different substrates (chemical patinas), additional additives, longer ageing, and varying climates.

In general, coatings #1 and 2 show that BTA improves coating performance, as seen by the difference in pitting corrosion, adhesion, and electrochemical impedance values. BTA does appear to affect solubility, as the coating without the additive is much more reversible before and after ageing. The ESBO plasticizers performed best and G-62 (coating #4) provided better scratch resistance, greater solubility after ageing, easier application, and improved adhesion compared to coating #1 with G-60. This product may be worth exploring in other variations. Matting agents appear to play an unexpected role in this study, as the matte Incralac (coating #6) and satin Permalac (coating #9) performed better by having the least corrosion, best electrochemical impedance values, and best appearance; however, the addition of the matting agent appears to reduce its solubility over time.

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MATERIALS LIST

1-methoxy-2-propanol Sigma-Aldrich Corp. St. Louis MO, USA www.sigmaaldrich.com/united-states.html

220 Bronze Sheet; Scotch-Brite™ Ultra Fine Hand Pad 7448 McMaster-Carr Santa Fe Springs CA, USA www.mcmaster.com/

Adhesion Tape, PA-280630 Gardeo, Inc. Pompano Beach FL, USA

CERAMAT 258; BYK-4513 BYK-Chemie GmbH Wesel, Germany

Benzotriazole and Petroleum ether Fisher Scientific Waltham MA, USA

Incralac products, Paraloid™ B-44 TALAS Brooklyn NY, USA

Paraplex® G-62, G-60, and A-8000 Hallstar Chicago IL, USA

Permalac® Peacock Laboratories, Inc. Philadelphia PA, USA

Qpcard 201 (card for digital camera management) Helsingborg, Sweden www.qpcard.com/

INSTRUMENTATION

PGSTAT204 potentiostat with a FRA32M frequency response analysis module (Metrohm, USA). Data were acquired over a frequency range of 100 mHz to 10 kHz (0.1 V signal amplitude), controlled by Nova software. A three-electrode system in a Paint Test Cell (Gamry Instruments, USA) was used. The set-up consisted of an Ag/AgCl reference electrode (AccumetTM), carbon counter electrode, and 0.1 M sodium sulfate electrolyte.

RhopointTM IQ goniophotometer 20/60/85° (Rhopoint House, UK). Three readings in a vertical, diagonal, and horizontal position were taken from each coupon; diagonal readings were the most regular, likely due to the sanding grain, and are provided in Table 2.

Bruker Hyperion 3000 FTIR microscope. ATR-FTIR analysis was performed using a dedicated $20 \times$ ATR objective containing a 100-µm germanium crystal, equipped with a liquid nitrogen-cooled narrow-band MCT-A detector. The sample area was defined using knife-edge apertures set to approximately 80×80 mm. The collected spectra are the sum of 64 scans at a resolution of 4 cm⁻¹.

Wolf-Wilburn Pencil Hardness Tester (BYK Additives & Instruments, Wesel, Germany).

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