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A. E. Salo and N. B. Garden

February 1959

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ABSTRACT

A spot-test procedure is outlined for preliminary selection of radiological-corrosion-resistant materials for use in chemical experiments.

SPOT TESTS AND RADIOLOGICAL CORROSION*

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Whenever radioactive isotopes are involved in chemical operations, it is essential to specify that the materials and equipment involved should have maximum resistance to the corrosion and radiation conditions to be expected in the particular application contemplated. Construction materials are often assaulted by high concentrations of acids, caustics, and organic solvents at the same time that they are subjected to high-level radiological conditions. The use of materials or equipment with questionable resistance to the conditions to be encountered can lead to a costly failure during operations or can result in damage to the equipment so that its usefulness is impaired or its reclamation is impossible because it cannot withstand the necessary decontamination procedures.

Unfortunately, the literature describing corrosion-resistant materials is often inadequate as a specification for the specialized applications such as the Health Chemistry Section of the Lawrence Radiation Laboratory is called upon to design for safety and efficiency. Few laboratories can afford to spend the required time to test completely even a fraction of the hundreds of products available on the market. To help our design engineers decide which products have potential merit, a spot-test procedure has been devised which has proved of great value in quickly eliminating some materials from consideration.

Test panels of a material under consideration may be submitted by a factory representative, or they may be prepared in the laboratory shops following the manufacturer's instructions. The procedure first involves dividing the surface of the test panel into twenty numbered squares, by scratching the surface with a sharp instrument or by marking it with an inked rubber stamp. One drop of each of the eighteen test reagents listed in the report form (Table I) is deposited on a square on the test panel. Each square is numbered corresponding to the reagent number on the report form. Squares numbered 19 and 20 are reserved for special reagents.

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*Work done under auspices of U.S. Atomic Energy Commission.

The reagent list evolved over a period of several years. It is designed to be as compact as possible yet represent a fair cross section of corrosive reagents as encountered in the chemical laboratories. An effort was made to choose representatives of the most common generic types, such as strong mineral acids, ketones, and esters. In some cases other members of a generic series could be readily substituted for those listed, such as MEK for Acetone.

Reagents are allowed to contact the material for 96 hours unless they evaporate in less time. Evaporation may be slowed down by covering with watch glasses, or highly volatile solvents may be replenished from time to time.

Results are reported by entering a brief qualitative statement in the space provided on the report form. Since this spot-test method is primarily one of preliminary elimination, no effort is made to make quantitative statements. If a material shows promising characteristics, further tests are conducted. Immersion or cup tests may be used in which weight, hardness, or other pertinent changes are noted quantitatively.

After a material has successfully passed the chemical-corrosion tests, it may be tested for radiological corrosion. Radiological corrosion consists of two categories: (1) contamination-decontamination, and (2) irradiation.

The contamination-decontamination spot test involves purposefully contaminating the surface with about 1 microcurie per square inch of any high-specific-activity radio nuclide. At LRL an actinide is used because it is readily available, and it is a fair representative of a series of elements likely to cause the most serious contamination problems. Nuclides such as phosphorus 32 or strontium 90 could, of course, give entirely different results, and tests with these nuclides are made if their presence is contemplated in future uses of the material under consideration.

The contaminating nuclide is deposited in solution form at about 1 N nitric acid concentration. After 1 hour contact in the acid state, an excess of ammonium hydroxide is added. The solution and resulting precipitate, if any, is dried for 24 hours at room temperature. To make the contamination test, a count is made of the radioactivity to determine the initial activity.

The surface is then washed with distilled water, wiped, air dried, and recounted to constant level. Results of the contamination test are reported as percent of initial activity retained. That portion of the radionuclide which is easily removed with water is not considered true contamination. The percent retained gives an indication of the propensity of a material to pick up and cling to radioactivity.

That portion of the nuclide which is not easily removed by water is considered true contamination and becomes the initial activity for the subsequent decontamination test. In the decontamination test, a series of washes is made with these reagents and in this order: 10% Dreft, 10% citric acid, 10% nitric acid. The samples are washed, rinsed with distilled water, wiped and counted to constant level between each reagent test. These reagents are considered to be non-destructive. If destructive reagents are permitted, all materials become decontaminable to a degree impossible to evaluate.

Until such time as a universal standard method of reporting is adopted, results of the decontamination test may be reported either as (1) percent retained, (2) decontamination factor in which

$$DF = \frac{\text{Initial Activity}}{\text{Final Activity}} ,$$

or (3) decontamination index where

$$DI = \text{Log}_{10} \frac{\text{Initial Activity}}{\text{Final Activity}} .$$

Which form is used here at LRL depends largely upon how familiar the engineer requesting the data is with these notations. Any one of the three forms, however, can be readily converted to any of the others.

Tentative minimum acceptance standards at LRL have been set, somewhat arbitrarily, as follows:

Reagent	% Retained
distilled water	10
10% Dreft	10
10% citric acid	1
10% nitric acid	0.1 .

Experience over the past several years has indicated that materials which do not meet these minimum specifications will be difficult, if not impossible, to decontaminate under actual field conditions.

Irradiation tests involve placing samples of the material in a 2000-curie Co^{60} source delivering a dose rate close to 10^7 roentgens per hour for various total exposures up to 10^9 roentgens. Qualitative effects are noted after different exposure periods. Chemical spot tests after radiation may then be made and compared with those before exposure.

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Table I

Report Form

To:

From:

Re:

Date:

Summary of Test:

Sample Preparation:

Testing Procedure: Surface was washed with warm, soapy water and dried. Area was marked off in suitable squares and a drop of each reagent below allowed to remain for 96 hours unless reagent was evaporated in that time. Surface was again washed with warm, soapy water and dried for inspection.

REAGENT	RESULT
1. Surfuric, 95%	
2. Nitric, 69%	
3. Hydrochloric, 36%	
4. Hydrofluroic, 48%	
5. Phosphoric, 85%	
6. Acetic, 99%	
7. Sodium hydroxide, sat'd.	
8. Sodium carbonate, sat'd.	
9. Ammonium hydroxide, 28%	
10. Chlorox (comm. strength)	
11. Gasoline	
12. Ethyl alcohol	
13. Acetone	
14. Ethyl acetate	
15. Carbon tetrachloride	
16. Benzene	

Table I

Report Form (cont'd)

Testing Procedure: (cont'd)

REAGENT	RESULT
17. Toluene	
18. Cleaning sol'n (chromic-sulfuric)	
19.	
20.	

"The above test conditions serve to compare various materials, when subjected to the same insult. The chemical effects noted are valid only for the conditions of the test. The results to be expected under other conditions can not be extrapolated from this test."

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