TECHNICAL NOTES

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

No. 411

RAPID CHEMICAL TEST FOR THE IDENTIFICATION OF CHROMIUM-MOLYBDENUM STEEL AIRCRAFT TUBING

By John C. Redmond
Bureau of Standards

Washington
March, 1932

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE NO. 411

RAPID CHEMICAL TEST FOR THE IDENTIFICATION OF CHROMIUM-MOLYBDENUM STEEL AIRCRAFT TUBING

By John C. Redmond

Reference was made in the National Advisory Committee for Aeronautics' Technical Note No. 350 (reference 1) to the desirability of a spot test, similar to the well-known test for nickel, for distinguishing between aircraft tubings made of plain carbon, and chromium-molybdenum steels (Society of Automotive Engineers (S.A.E.), 1025 and 4130 X steels). Attempts made in this laboratory to develop such a test have been unsuccessful. However, a simple, rapid, qualitative test which can be applied to solutions of drillings or chips has been devised. The test is based on the orange-red compound which is formed when thiocyanate and quinquevalent molybdenum react. This test is much more reliable than the potassium ethylxanthate test which has been recommended for a like purpose.* A list of the apparatus and reagents which are required, and a description of the procedure follow. If desired, the reagents and apparatus can be assembled in a simple portable kit.

1. Apparatus required:

(a) one test tube 1" x 8", for each sample.
(b) one No. 5 rubber stopper for each test tube.
(c) one dipping pipette to deliver 10 ml.
(d) one dipping pipette to deliver 5 ml.
(e) one dipping pipette to deliver 2 ml.
(f) one small porcelain spoon (99 mm, capacity approximately 0.4 ml).
(g) one container of boiling water in which the test tubes may be heated to hasten the solution of the samples. A 600 ml low form beaker is a convenient size.

---

* Private communication from W. J. Jeffries, Beaver Falls, Pa.

2. Reagents required:

(a) Concentrated hydrochloric acid (HCl), (sp.gr. 1.19).

(b) Potassium chlorate ($KClO_3$), crystals, reagent quality.

(c) Stannous chloride ($SnCl_2$) solution. Dissolve 125 g of $SnCl_2 \cdot 2H_2O$ in 100 ml of hydrochloric acid, and when solution is complete add 100 ml of water. This solution deteriorates and should be renewed at least every six months

(d) Potassium thiocyanate (KCNS) solution. Dissolve 50 g of KCNS in 100 ml of water.

(e) Ether, reagent quality. (While this reagent is not necessary for the test it is sometimes useful, as described later.)

3. Procedure:

Transfer 0.2 g (approximately) of drillings to the test tube. Place the test tube in the vessel of boiling water, and add from a pipette (c) 10 ml of concentrated HCl. After two minutes add with the spoon approximately 0.3 g (a heaping spoonful) of $KClO_3$. If the sample is not dissolved within eight minutes a second spoonful of $KClO_3$ should be added. When solution is complete remove the test tube from the water bath, cool, and add 15 ml of cold water. Then add from a pipette (d) 5 ml of $SnCl_2$ solution, stopper and shake the test tube. The amount of chromium present may be estimated at this point by the depth of green color of the solution. Finally add from a pipette (e) 2 ml of KCNS solution, and again stopper and shake the test tube. The appearance of an orange-red color indicates the presence of molybdenum, the amount of which may be estimated by the depth of color of the solution. The test will easily show the presence of 0.05 per cent of molybdenum.

The colored molybdenum compound may be obscured to some extent by the other colored compounds that are originally present in the solution of the steel. In addition, it is sometimes necessary to test steels of which less than 0.2 g of sample is available or in which the molybdenum content is less than that of S.A.E. 4130 X. In such cases, it is of advantage to extract the colored molybdenum compound with ether. To do this, add 5 ml of ether to the cooled solution, stopper the test tube, and shake vigorously. Set aside until the ether separates. This is the upper layer and contains the orange-red molybdenun-thiocyanate compound, but none of the other colored compounds. The color is deepened by virtue of the concentration of the colored molybdenum compound in the smaller volume.

With a little practice an inexperienced operator may weigh out and test six samples in approximately twenty minutes. While the chemical test is not as rapid as the spark test, (reference 1 and 2) it is none the less of advantage when only occasional tests or checks on physical tests need be made. Although the test requires filings or cuttings, it is more or less nondestructive as regards the material to be tested, for the small amount of sample required may be taken from the ends of the tubes. With care it can be applied to built-up aircraft structures by carefully filing the extreme end of the tube to be tested, or by lightly filing enough of the surface area.

Bureau of Standards, Washington, D. C.
February 1, 1932.

REFERENCES

1. Mutchler, W. H., and Buzzard, R. W.: Methods for the Identification of Aircraft Tubing of Plain Carbon Steel and Chromium-Molybdenum Steel" T. N. No. 350, N.A.C.A., 1930.

2. Hildorf W. G., and McCollam, C. H.: Practical Aspects of Spark Testing. Iron Age, 124, 953 (1929).