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*File: Quantitative Spectrochemical Analysis of Nickel Cathode Surfaces*

Title **Quantitative Spectrochemical Analysis of Nickel Cathode Surfaces**

By

**Electronic Tube Engg.** Div.

Information prepared for **Electronic Tube Engg. Div.**

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Date **3-25-60**

*CWA CMJ WMS VC  
PES YRS PGM PM  
JMS,  
RMBE RMB  
GLC or*

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Quantative Spectrochemical Analysis  
of Nickel Cathode Surfaces

Summary:-

At the request of the Ken-Rad Division, work was undertaken in cooperation with Schenectady Works Laboratory to find a rapid spectrographic method for analyzing radio tube cathode slaves for small amounts of impurities. It is now possible to analyze for Si, Mg, In, Cu, and Fe within a range which is useful for some cathode base materials.

Method:-

Cathodes are received at the laboratory with the surface in the condition desired by the Ken-Rad Division for the analyses. That is, the cathodes may either be sampled by Ken-Rad as received or may be cleaned or etched in any way the Division desires for the particular study to be made.

The cathodes are in the form of small cylinders (about 1" long by 1/16" diameter with about .005" walls). These are flattened by pressing against a clean glass plate - making sure, in the case of lock-seam cathodes, that the seam is not next to the glass plate. The flat cathode is then bent over a carbon spectrographic electrode of 1/4" x 1/4" cross section and held securely by a small alligator clip. The surface flattened against the glass is the one exposed for subsequent sparking.

This mounting is used as the lower electrode in a spark gap light source for a medium quartz prism Zeiss spectrograph.

The spark source is a "Feussner" working from a 110 volt regulated line and giving a working voltage of 15,000. A rotary interrupter is used and the spark settings are:

Capacitance  $1.333 \times 10^{-3}$  microfarads; Inductance  $8 \times 10^{-5}$  herrys. The timing consists of 15 second prespark in all cases with a 1 minute exposure for the Inco standards (solid rods of analyzed nickel) and 75 seconds for samples. The electrodes are spaced 2 mm. apart and the upper one is a pointed spectrographic carbon electrode.

Spectrographs are made on 10 inch plates and analyzed in the densitometer. All of this work is now done in the Schenectady Works Laboratory Spectrographic Section.

Results:-

Between August 1, 1945 and January 7, 1946 over 125 spectrograms have been analyzed. The laboratory is now prepared to handle an average of 10 samples per day for Ken-Ra<sup>®</sup> and to give results for:

Fe between 0.02%	-	0.5%
Mg between 0.01%	-	0.5%
Si between 0.15%	-	1.5%
Mn between 0.02%	-	1.0%
Cu between 0.09%	-	1.0%

These analyses give quantitative results for only the above elements (for which standards are available), and only for the surface exposed.

Discussion:-

1. The spark is estimated to remove only about 0.1 mil of nickel. Consequently, the analysis is truly a "surface analysis" and the results reported for the impurities may differ by a factor of 3 to 5 from the total analysis of the nickel cathode. However, there are those who believe that, despite migration of impurities in the nickel during life, the surface analysis is the most important for determining emission.
2. The silicon sensitivity is not all that is to be desired, but with the present method and samples there is little hope of much improvement. The sample size is far from ideal for any quantitative analysis and the disadvantages of this sample size are apparent in this difficulty.
3. Iron analysis results have been consistently erratic - varying with individual cathodes in a single lot and probably along a single cathode. The variation between spectrographic surface analysis and total analysis has also been great (sometimes 7 to 1). However, it is believed that the analysis variations do give a true picture of what occurs.
  - a) Iron is usually "high" on a drawn or rolled nickel surface for it is "picked-up" from the dies and rolls. This has been confirmed by other companies.
  - b) The iron deposited on the surfaces is not in a uniform layer but only in spots. This has been checked by electrographic means by Bell Labs.
4. Gross spectrographic analyses of nickel cathodes are

not generally available at Schenectady Works Laboratory at this date; but one standard is being set up and, if the acquisition of three or four more standards becomes possible, a method may be instituted in the future using a 100 mg. pellet and a d-c arc.

R. E. Roth

*Raymond E. Roth 3/20/46*

Countersigned by:

*KC DeWalt March 25, 1946*

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