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INVESTIGATION OF AN ION-EXCHANGE SEPARATION PROCEDURE

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This report briefly describes the extension of a previous investigation (1) of the anionic behavior of metals with an ion-exchange resin -- HCl system. In both instances, an attempt is being made to adapt an idea of Hicks, et al., (2) for use as a separations method in a qualitative analysis scheme based upon radioactivation analysis.

A few preliminary experiments were made. However, this approach was discarded because one of the participants (M. M. Vick) did not have sufficient time to properly apply his services to the problem. Thus, in order that some information might be obtained on a method that seemed to be applicable, certain samples received as part of the Activation Analyses Group's service program were analyzed.

Among these materials were several catalysts, used in cracking petroleums, which had a complex trace element spectrum by spectrographic methods. The trace elements in each sample varied with the catalytic action of the sample. One catalyst (sample D) was at peak catalytic action; a second (sample E) had been used in the cracking process and still retained appreciable catalytic activity; and the third (sample F) was a used catalyst which had lost its catalytic usefulness.

Each sample was irradiated for two weeks in the LITR (flux = $\sim 10^{14}$ neutrons/cm²/second) and after the irradiation was dissolved in 12 M HCl and dispersed on a Dowex 1 resin column. The column bed was 10 cm long and 0.4 cm in diameter and was packed with 200-400 mesh resin. After the sample dispersion, elution was accomplished with equal volumes (25 milliliters) of 12 M HCl, 6 M HCl, 3 M HCl, 1 M HCl and water. Each fraction was either aliquotted to, or evaporated to a smaller volume and then aliquotted to a

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sample mount and the radioactivity assayed. The resin was slurried from the glass column and treated in a similar manner. In each instance, the radioactivity assay included measurements for half-life (decay), beta activity (aluminum absorption), and gamma activity (scintillation spectrometer). One fraction, the 12 M HCl fraction contained so many different radioactivities that it was necessary to use the method proposed by Handley⁽³⁾ to separate it further.

Table I reports the results of the assay of these catalyst samples. In those instances where an element is reported as "probably present", very little activity was observed in the conditions used to assay the radioactive sample.

Table I

Qualitative Detection of Trace Elements in Catalytic Cracking Agents
by a Radioactivation Analysis

<u>Radioelement Found</u>	<u>Sample D</u>	<u>Sample E</u>	<u>Sample F</u>
45			
Ca60	P	P	P
Co	PP	PP	P
Cr51	P	P	P
Cu64	PP	PP	P
Fe59	P	P	P
Ga72	PP	P	P
Hf181	P	O	PP
Ir192	P	P	P
Na24	P	P	P
Ni58 (as Co ⁵⁸)	PP	PP	PP
P32	P	P	P
Pt197	PP	PP	PP
S35	O	PP	PP
Sb122	P	P	P
Sc46	P	P	PP
Si31	P	P	P
Sr85	P	P	O
W187	P	P	P
Zr97	P	P	P
Zn65	P	P	P

P = Present; PP = Probably Present; O = Absent

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All of the radioelements, except Co^{58} , are the products of the thermal neutron capture reaction, n, γ , and it can be assumed that the stable isotopes of these elements were present in the sample. In the instance of Co^{58} , an n, p reaction on Ni^{58} would produce Co^{58} , it can be assumed that nickel might also be present in each sample. Gross amounts of Na^{24} radioactivity, arising from either the n, γ reaction on natural sodium or from the n, p reaction upon aluminum (a major sample component), made it advisable to wait three days before beginning the analysis. Thus, the table does not include those elements in which only a short half-lived radioisotope can be induced. It would be necessary to make specific separations for these elements.

Table I indicates that there was very little variation in the elements detected in the three catalysts. However, a build-up of radioactivity on samples E and F indicated an accumulation of contaminants. Quantitative analyses by usual activation analysis methods could determine the extent of this contamination.

On the basis of this preliminary work, it was believed that some evidence of the distribution of certain elements in this scheme had been obtained. It was as follows:

<u>Fraction</u>	<u>Element Observed</u>
12 M HCl	As, Ca, Cr, Hf, Na, Ni, P, S, Sc, Si, Sr
6 M HCl	Cr, Cu, Hf, Na, P, Sc, Zr
3 M HCl	Co, Cr, Cu, Fe, W, Zr
1 M HCl	Fe, Ga, Sb, Zn
H_2O	Fe, Pt, Zn
Resin	W, Ir

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There is an overlap of some ions in the various fractions. This is probably due to too small a ratio of eluant to column volume ($\approx 4:1$).

There was no indication of valence state of S or P.

Further work is intended in which the distribution of trace quantities of the various elements throughout the different elution fractions from an anion resin -- HCl system will be followed more completely. In addition, systems involving HBr and HI will be studied.

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 2. H. G. Hicks, R. S. Gilbert, P. C. Stevenson, and W. H. Hutchin, "The Qualitative Anionic Behavior of a Number of Metals with an Ion Exchange Resin," Dowex 2, IRL-65, Calif. Research and Development Company (December, 1953).
 3. T. H. Handley, "Qualitative Analysis Scheme," Anal. Chem. Quar. Prog. Rep., Jan. 10, 1951, ORNL-955, p. 47.

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