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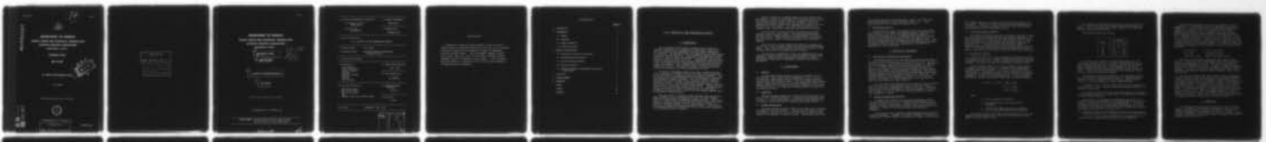

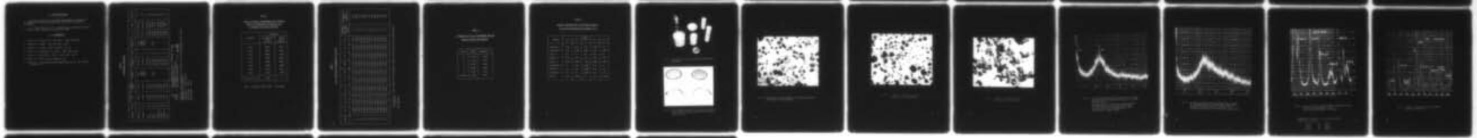

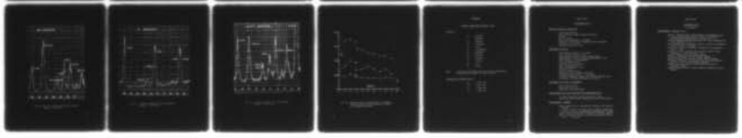

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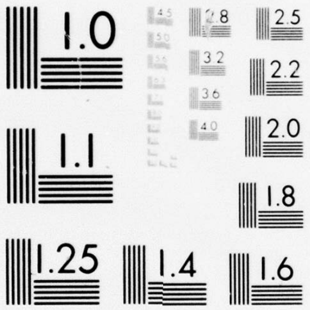

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TECHNICAL NOTE

MRL-TN-388

X.R.F. ANALYSIS OF SOME REGENERATED CATALYSTS

W. B. Searle*

*Materials Research Laboratories, N.S.W. Branch

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ABSTRACT

Demands for a rapid and accurate analysis of some of the trace elements present in regenerated catalysts has led to the use of a grinding-briquetting technique for the preparation of samples of regenerated petroleum cracking catalysts. The trace elements vanadium, chromium, iron, nickel, copper, zinc, lead, the rare earths, lanthanum, cerium, neodymium, may all be determined on the one sample. A more informative empirical formula for evaluating the level of trace metal contamination in regenerated stock is proposed.

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X.R.F. ANALYSIS OF SOME REGENERATED CATALYSTS

1. INTRODUCTION

The two amorphous silica-alumina cracking catalysts commercially available, designated as a low alumina and a high alumina, contain 9-12% and 24-25% aluminium oxide respectively, and both contain approximately 9-12% moisture. The moisture content is of importance as the fresh catalyst is sold on a moisture free basis. After ignition at temperatures ranging from 900°F in the cat-cracker and regenerator, the moisture content of the catalyst is reduced to about one per cent. The molecular sieves (synthetic zeolites) incorporated in the fresh feed are commercially designated as X or Y type, and contain different concentrations of the rare earths, lanthanum, cerium, and neodymium. Their stronger acidity is reported to generally increase the activity of the catalyst and improve the conversion process, producing more gasoline, less coke, gas and heavy cycle oil.

The metallic contaminants sodium, iron, nickel, vanadium and copper, cause a decrease in the catalytic activity and result in the decomposition of the hydrocarbons to hydrogen and coke at the expense of gasoline. Lead, entering the feed through contamination with tetraethyllead or tetramethyllead permanently poisons the catalyst. The loss in gasoline production resulting from cracking catalyst of low selectivity can cost a refinery thousands of dollars. Catalyst addition rates may range from 1/2 ton per day to around 5 ton per day on a fresh catalyst charge. However, this also depends upon the coke-burning capacity of the unit (1). Acceptable guidelines for evaluating the level of contamination may be established by determining the concentrations of some of the trace elements commonly found in regenerated cracking catalysts.

This paper reports a relatively simple grinding-briquetting method of sample preparation for the determination of some of the trace elements present in current samples of regenerated cracking catalysts. The need for this X.R.F. technique has arisen from the difficulties encountered by using common wet chemical techniques, and also from the need to monitor the activity of cracking catalysts by the petroleum refinery. A more informative empirical formula for evaluating the level of trace metal contamination in the regenerated stock is presented.

Numerous techniques for preparing samples for X.R.F. analysis have been proposed. Rayburn (2) and Townsend (3) have described fusion methods. The lithium tetraborate straight fusion and the lanthanum oxide heavy absorber fusion methods of Rayburn are time consuming. Further, his lanthanum oxide fusion technique would complicate the analysis of the rare earths content of the catalyst. The sodium borate fusion method of Townsend (4) may suffer from cracked discs and is also time consuming.

Stone and Rayburn (4) have described a grinding technique using polyvinyl alcohol as a pellet binder. They have obtained reasonable agreement with the chemical analysis for the determination of the total rare earth content. However, Rayburn (2) indicates that this method may give unsatisfactory results for the determination of iron, nickel and vanadium.

Dryoff and Skiba (5) have analysed used catalysts by measuring the net intensity of the fluorescent radiation emitted from an unpacked ground powder. Their observations have shown a significant variation at the 95% confidence limits, due to the packing density variations.

The quantitative determination of the trace elements present in the regenerated cracking catalyst, using a simple grinding-briquetting sample preparation technique, and the evaluation of the degree of catalyst contamination, which may be of some general interest, forms the basis of this report.

2. EXPERIMENTAL

2.1 Samples

The regenerated alumina catalysts analysed were sampled from five local sources. These sources have been designated as M, N, O, P and Q. The fresh feed alumina catalyst used to prepare the standard samples was obtained from a local commercial source. Over fifty samples from the above sources received during 1974 and 1975 have been analysed for several trace elements together with the analysis of some of the samples for rare earths. The moisture content of the regenerated cracking catalysts was found to be less than one per cent.

2.2 Apparatus

The X-ray equipment employed was a Philips PW 1540 fluorescence unit and a PW 1010 (1 kilowatt) generator. The operating conditions are listed in Table 1. A pulse height analyser was used for all the determinations. A helium path was also available for analysing loose powder samples.

2.3 Sample Preparation

The fresh catalyst was dried at 500°C for four hours prior to grinding and mixing in a Pitchford Ball Mill. Preparation of the sample required the addition of two grams of boric acid to five grams of catalyst and the milling of the components for about 10 minutes. Approximately three grams of the ground sample were then briquetted at a load of 6 x 10^3 kg, into a 32 mm biscuit using the tool steel die shown in Fig. 1 (6). Typical biscuits prepared by this technique are shown in Fig. 2.

2.4 Calibration Curves

Several duplicate pellets were prepared from the pure oxide and the fresh catalyst for each element, using weight dilution, covering the range of zero to about 1.5 per cent. The analytical working curves were established by plotting the peak to background ratio, and also by plotting the ratio of the analyte line intensity to the line intensity from an external standard containing the element versus the weight per cent. These two separate calibration lines were obtained by the least squares procedure. The latter was found to give better repeatability of intensity ratio measurements on a day to day basis.

3. RESULTS AND DISCUSSION

3.1 Evaluation of Particle Size Effects

The average particle size of the regenerated catalysts received was in the order of fifty micrometres (see Fig. 3), while that of the fresh feed varied from about ten to one hundred micrometres, (see Fig. 4). Agglomerates of a (0-5) micrometre sample of ground regenerated catalyst are shown in Fig. 5. To estimate the effect of grain size different particle size fractions from a sample of fresh high alumina catalyst, containing ten per cent of Y type molecular sieve were analysed for lanthanum. The observed lanthanum intensity from each fraction of an unbonded, loosely packed sample was then compared with the same sample ground to (0-5) micrometres. The vanadium intensity was also measured from samples in the 'as received' condition and compared to the same sample ground to less than five micrometres and these results are shown in Table 2.

All of the observations showed about two per cent increase in intensity relative to the (0-5) μm ground sample, however, duplicate readings on (0-5) μm samples gave a repeatability of about 0.5 per cent. The average particle size of the ground samples and calibration standards was less than five micrometres. Grain size effects in the ground samples of catalysts were considered to be minor.

3.2 Optimum Briquetting Pressure

The analyte line intensity for each element analysed was found to increase about seven per cent for an increase in the briquetting pressure from zero to about 2 x 10^3 kg load. However, beyond 3 x 10^3 kg load, the intensity variations arising from fluctuations in the applied pressure were negligible. A load of 6 x 10^3 kg was found to give reproducible intensities.

At the pressure used a sufficiently dense and smooth surface layer was obtained consistently. Although it is usually preferable to evacuate the pressing tool to remove enclosed air occluded in the sample, no deterioration of the sample surface was noted after the sample had been evacuated in the spectrometer. Briquettes of fresh catalyst showed some deformation when the briquetting pressure was released, on the other hand tablets prepared from regenerated catalysts showed high stability.

3.3 X-ray Diffraction Analysis

X-ray diffraction data for both types of molecular sieve was consistent with the presence of a sodium aluminium silicate hydrate (faujasite). An X-ray diffractogram for a sample of fresh feed catalyst containing approximately five per cent of Y type molecular sieve is shown in Fig. 6. The two intense peaks are the (III) and the (533) diffraction lines from the molecular sieve. A diffraction pattern obtained from a sample of regenerated catalyst containing one per cent of molecular sieve is shown in Fig. 7. These graphs show that quantitative X-ray diffraction analysis of samples containing low concentrations of molecular sieve would be unsuitable.

3.4 Analytical Results

The total concentration of the trace elements in regenerated catalysts rarely exceeded two per cent. Samples containing more than one constituent, e.g.vanadium and nickel up to 0.5% were compared to the original calibration standards. The results indicated that interelement effects were minor.

X-ray fluorescence scans of X and Y type molecular sieves indicated the presence of several rare earths: lanthanum, cerium, praseodymium, neodymium, samarium and europium. An X.R.F. scan of 10% Y type sieve is shown in Fig. 8. The presence of these elements complicated the analysis, in particular the chromium Ka line had to be corrected for the overlapping lanthanum Lb, cerium Lb and the vanadium Kb lines. Thus the following mathematical correction was applied to obtain the true chromium intensity :

$$\begin{aligned} I_t\,(\mathrm{Cr\ Ka}) = I_o\,(\mathrm{Cr\ Ka}) &- 0.088\ I_n\,(\mathrm{VKa}) \\ &- 0.12\ \ I_n\,(\mathrm{LaLa}) \\ &- 0.035\ I_n\,(\mathrm{CeLa}) \end{aligned}$$

where

I_t - the true analyte line intensity

I_o - the observed analyte line intensity minus background

I_n - the difference between the true line intensity for the interfering element and the background.

The correction factors were found by obtaining the ratio of the overlapping Kb (or Lb) emission line to the Ka (or La) emission line using a sample of the pure metal or oxide.

Two substandards containing five per cent of X type sieve and ten per cent of Y type sieve with the balance of high alumina fresh catalyst were prepared. These substandards were designed to conform to the commercial blends available at the present time.

The following results were obtained :

Element	Sub Standard	
	5X	10Y
%La	0.405	0.722
%Nd	0.123	0.243
%Ce	0.063	0.097

The average sieve contents for thirteen samples based on a "10 Y" composition, are shown in Table 3. Typical recorded traces from M, N, O and P regenerated catalysts are shown in Figs. 9-12 and cover an angular range of approximately 20^{o} (2θ). Results obtained for the various elements present in thirteen samples of regenerated catalyst are shown in Table 3. Sodium could not be determined as it was outside the scope of the X-ray fluorescence instrument available.

Characteristic trends in trace elements in the regenerated catalyst were also evident and shown graphically in Fig. 13. Eleven consecutive samples of M catalyst, sampled over several months, indicated similar variations in the concentrations of nickel, vanadium and lead.

A comparison of X.R.F. and wet chemical data for the determination of vanadium, given in Table 4, indicates satisfactory agreement between the two methods for this element.

The analysis of most of the other trace metals present in the samples of regenerated catalyst was not attempted due to the difficulties encountered using wet chemical techniques.

The accuracy of the technique was determined by the quality of the standards prepared by weight dilution, and by measurement of the true line intensity, in particular, correcting for overlapping lines. The results from different briquettes of the same sample were within five per cent of each other. The coefficient of variation for the determination of vanadium colorimetrically and by X.R.F. was 4.5 per cent.

3.5 Empirical Formula for Evaluating the Level of Contamination

The levels of trace element contaminants in the regenerated stock are required so as to optimise the operation of the cat-cracking unit.

Several empirical formulas are available to determine the degree of contamination. A contamination index (C.I) formulated by the American Cyanmid Co. (7), may be calculated from the weight concentrations of the elements Fe, V, Cu and Ni, i.e. C.I. = 1000 (Fe + 4V + Cu + 14 Ni). Modifying this formulation a more comprehensive empirical index may be applied to emphasise the deleterious nature of other elements present : (C.I.) (modified) = 1000 (Fe + 4V + Cu + 14Ni + Cr + Zn + 10Pb). Values of C.I. (modified) have been calculated for thirteen samples and have been recorded at the end of Table 3. The terms of Cr, Zn, 10Pb have been added, as these elements can cause problems during the cat-cracking operation and detrimental properties in the refined product. A guideline for determining the degree of contamination may be evaluated using the following arbitrary index levels :

Less than 500	:	fairly clean catalyst
500 - 2000	:	contaminated catalyst
Greater than 2000	:	heavily contaminated catalyst

Referring to the calculated contamination index values shown in Table 3, it is seen that M and O regenerated catalysts may be considered to be heavily contaminated, and N, P and Q samples fall in the range of a fairly clean to contaminated catalyst. The latter contamination index gave an overall idea of the total level of impurities present in regenerated catalysts. The need for recycling or replacement of the catalyst may be assessed by monitoring the individual levels of vanadium, nickel, copper, lead and iron.

Over a period of about twelve months several samples of regenerated catalyst from source M were taken and the vanadium and lanthanum contents as well as nickel and lead, were determined. A significant decrease in the vanadium content from 0.15% to 0.08% was recorded. It was thought that some changes, possibly in the addition rate of the fresh feed to the cat-cracker had occurred. To supplement the requirements of the cracking catalyst as regards to selectivity, the carbon content may also be determined. On the regenerated samples of catalyst received the carbon content was in the order of 0.4%. The trace elements present do not give an indication of the carbon content of the sample as these may reside in an active or inert state. Nominal values of some of the trace elements found in regenerated catalysts from each source are shown in Table 5.

4. CONCLUSION

The grinding-briquetting method has been found to give satisfactory results. The samples received showed considerable variations in trace element contamination and in molecular sieve content. The need for recycling or replacement of contaminated or deactivated catalysts in current use may be determined by monitoring the levels of the major trace elements, vanadium, nickel, lead and iron.

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TABLE 1

INSTRUMENTAL CONDITIONS

Element	Line	Wavelength Å	Angular Position $2\theta^{o}$	Overlapping Lines	Analysing Crystal	Target	Correction Factors	L.L.D.	Path	Detector
Vanadium	Ka	2.505	76.92	TiKb	LiF 200	W	0.140	0.003	Vacuum	Fl
Chromium	Ka	2.291	69.34	VKb) LaLb) CeLb)	LiF 200	W	0.008) 0.120) 0.035)	0.002	Vacuum	Fl
Iron	Ka	1.937	57.49	-	LiF 200	Cr	-	0.002	Helium	Fl
Nickel	Ka	1.659	48.65	-	LiF 200	W	-	0.002	Vacuum	Fl
Copper	Ka	1.542	45.02	NiKb	LiF 200	Cr	0.004	0.002	Vacuum	Fl
Zinc	Ka	1.437	41.81	-	LiF 200	W	-	0.002	Air	Sc
Lanthanum	La	2.665	82.86	-	LiF 200	W	-	0.008	Vacuum	Fl
Cerium	La	2.561	128.11	-	LiF 220	W	-	0.009	Vacuum	Fl
Neodymium	Lb	2.370	99.02	-	LiF 220	W	-	0.009	Vacuum	Fl
Lead	La	1.175	33.92	-	LiF 220	W	-	0.009	Air	Sc

Fl. : Flow proportional counter, 10% Methane in Argon
Sc. : Scintillation Counter
Targets : W - Tungsten: Cr - Chromium.
L.L.D. : Lower limit of detection: $\text{L.L.D.} = \frac{3}{\text{Mav}}\sqrt{\frac{\text{Rb}}{\text{Tb}}}$

Where Mav. is the average slope in C/S/% : Rb *is the accumulated counts on the* background for a period Tb.

Collimation : Fine (160 μm)

Tube Voltage : 54 kV

Tube Current : 18 mA

TABLE 2

VANADIUM INTENSITY MEASUREMENTS FROM POWDERED SAMPLES OF REGENERATED CATALYSTS IN THE AS RECEIVED AND GROUND CONDITION

% Vanadium	Treatment	
	'As received' Net Intensity	Ground Net Intensity
0.153	98145	96297
0.143	92644	92519
0.101	69074	67991
0.052	39859	38462
0.035	28938	27019
0.028	23829	22884
0.014	13917	14122
0.003	7409	7368
Particle Size μm	30-60	< 5

Time to accumulate above counts : 100 seconds.

TABLE 3

X.R.F. ANALYSIS OF REGENERATED CATALYSTS

Source	% Cu	% Ni	% Fe	% Cr	% V	% LA*	% ND*	% Ce	% Zn	% Pb	Average % of Y type Molecular Sieve	Contamination Index (modified)
M	<0.005	0.038	0.86	0.016	0.153	0.273	0.072	0.039	0.018	0.051	3.6	2548
M	<0.005	0.034	0.80	0.012	0.133	0.284	0.078	0.044	0.015	0.045	3.9	2285
M	<0.005	0.032	0.67	0.011	0.110	0.310	0.086	0.038	0.011	0.059	3.9	2170
N1	<0.005	0.022	0.61	0.003	0.052	0.248	0.060	0.031	0.003	<0.009	3.0	1132
N1	<0.005	0.020	0.57	0.005	0.051	0.234	0.054	0.030	0.003	<0.009	2.9	1062
N1	<0.005	0.015	0.48	0.003	0.035	0.266	0.064	0.033	<0.002	<0.009	3.2	835
N2	<0.005	0.003	0.29	0.002	0.012	0.015	<0.008	<0.008	0.002	<0.009	<0.5	382
O1	<0.005	0.017	0.61	0.009	0.014	<0.008	<0.008	<0.008	0.018	0.217	<0.5	3101
O2	<0.005	0.008	1.12	0.005	0.010	<0.008	<0.008	<0.008	0.036	0.429	<0.5	5603
P1	<0.005	0.008	0.76	0.020	0.004	0.018	0.029	0.010	0.003	0.011	1.2	1017
P2	<0.005	0.009	0.81	0.023	0.005	0.076	0.018	0.009	0.004	0.009	0.9	983
P3	<0.005	0.008	0.76	0.018	<0.003	0.137	0.026	0.011	0.003	0.014	1.4	1033
Q	<0.005	0.017	0.29	0.005	0.029	0.326	0.093	0.037	0.003	0.009	4.1	651

The numbers 1, 2 and 3, refer to the different cracking units operated by the same source, N, O or P.

*See Appendix.

TABLE 4

A COMPARISON OF X-RAY FLUORESCENCE AND WET CHEMICAL RESULTS FOR VANADIUM

Source	X.R.F. % V	Chemical % V
M	0.153	0.148
M	0.141	0.151
N	0.052	0.054
N	0.049	0.050
N	0.035	0.034
Standard	0.020	0.016

TABLE 5

NOMINAL CONCENTRATIONS OF THE TRACE ELEMENTS IN CATALYSTS RECEIVED SINCE JANUARY, 1975

Element	Source				
	M	N	O	P	Q
Vanadium, %	0.08	0.04	0.01	0.005	0.03
Chromium, %	0.01	0.005	0.007	0.02	0.005
Iron, %	0.8	0.4	0.8	0.8	0.3
Nickel, %	0.04	0.02	0.01	0.01	0.02
Copper, %	<0.005	<0.005	<0.005	<0.005	<0.005
Lanthanum, %	0.3	0.24	<0.008	0.10	0.3
Cerium, %	0.4	0.03	<0.008	0.01	0.04
Neodymium, %	0.08	0.6	<0.008	0.02	0.09
Lead, %	0.05	<0.009	0.3	0.01	<0.009

FIG. 1 - Tool steel die used to prepare the briquetted specimens.

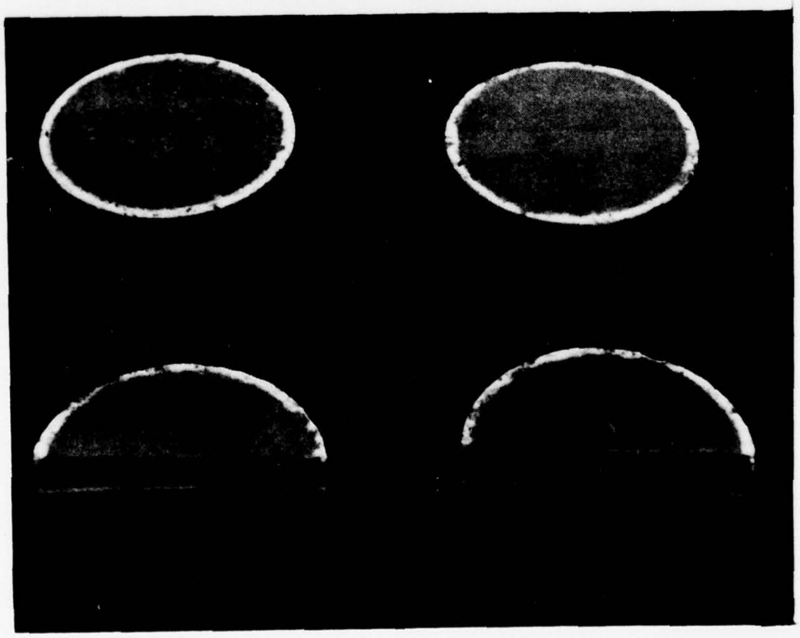

FIG. 2 - Two briquetted samples of regenerated catalyst with a boric acid case, and a section through another tablet.

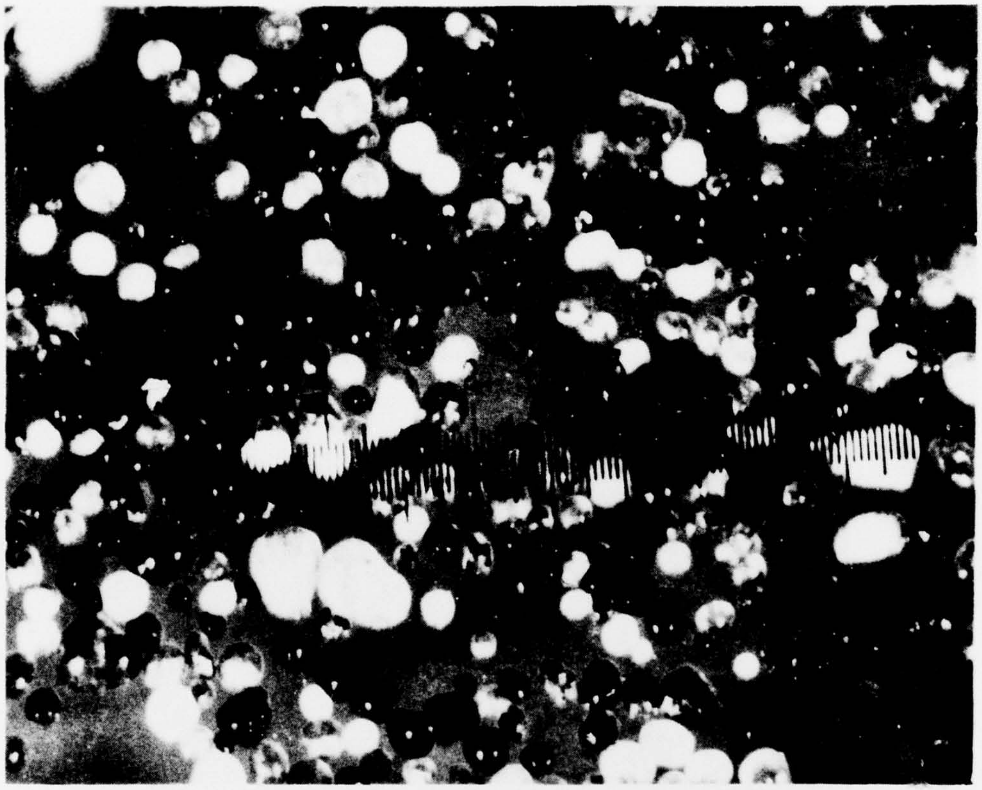

FIG. 3 - Particles of silica-alumina in a regenerated catalyst.
(1 Division = 10 Micrometres).

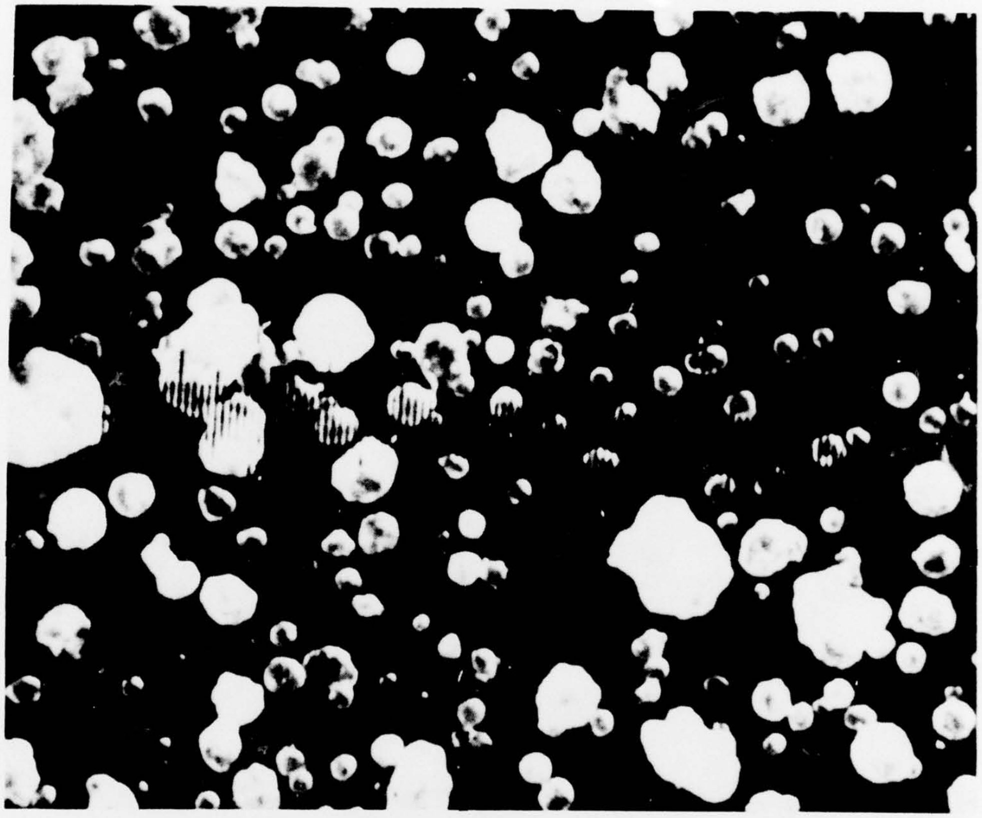

FIG. 4 - A sample of fresh feed catalyst.
(1 Division = 10 Micrometres).

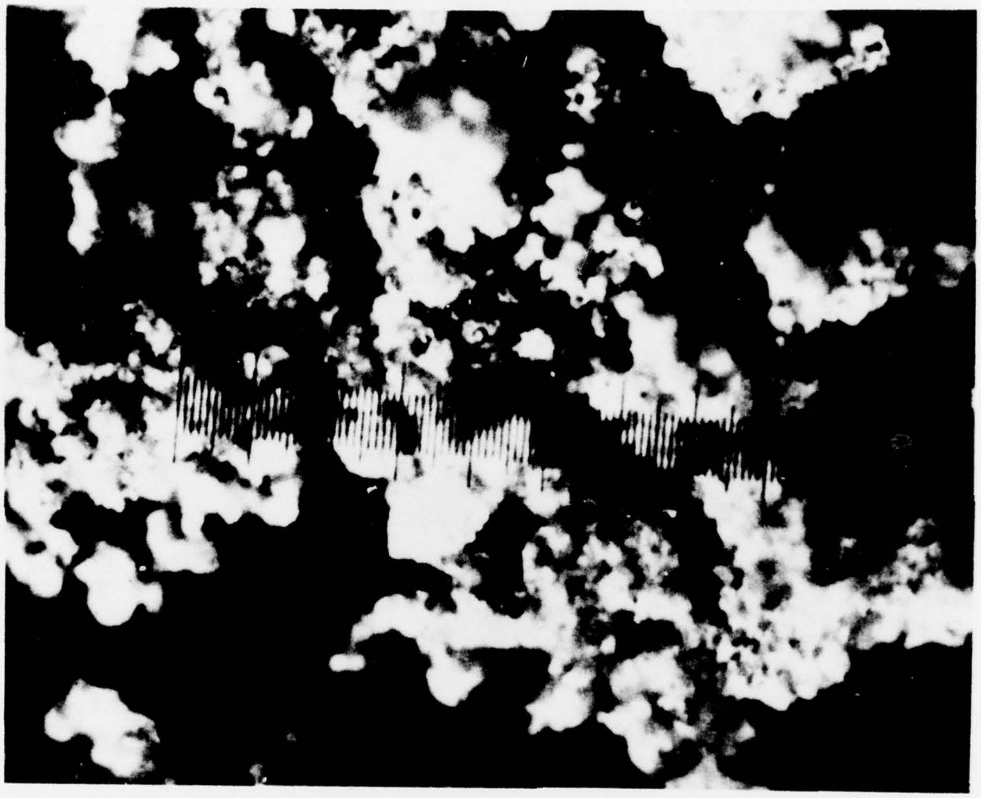

FIG. 5 - A sample of a ground catalyst.
(1 Division = 10 Micrometres).

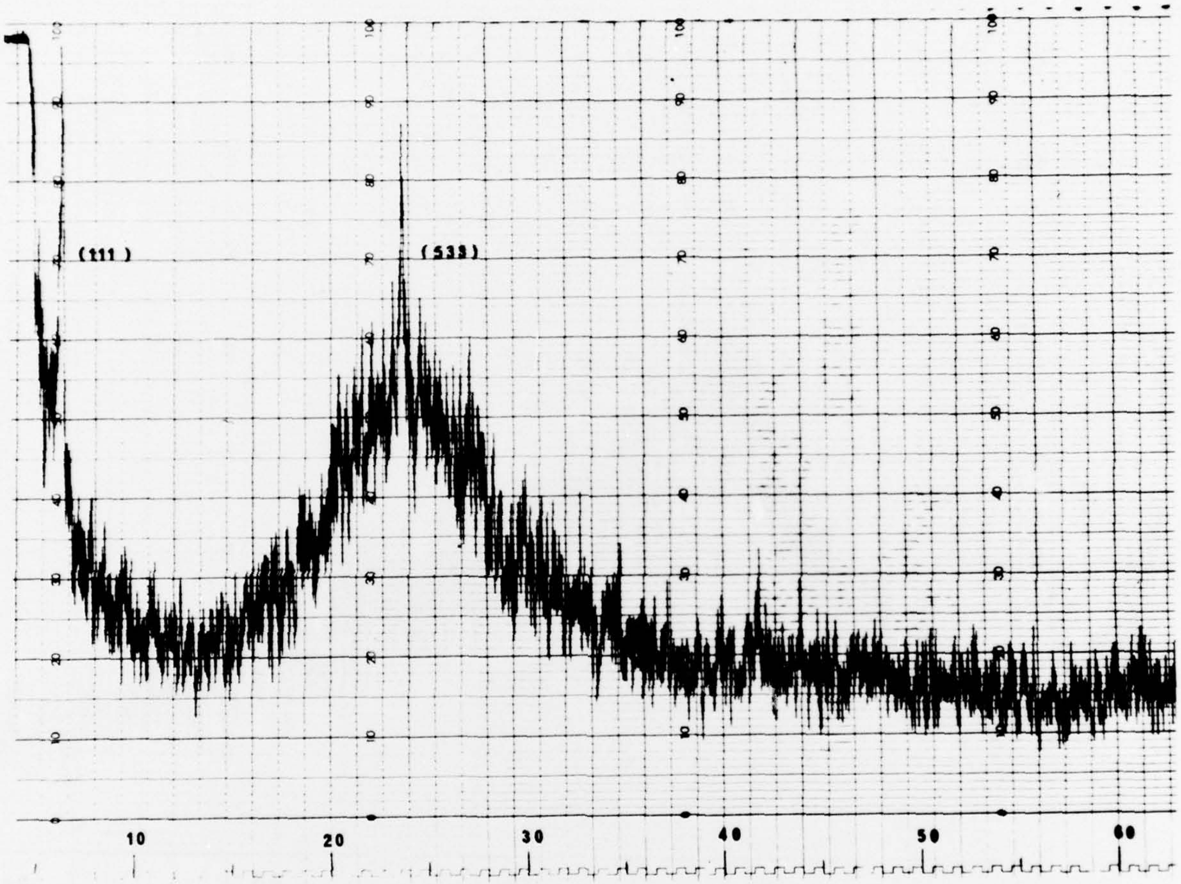

FIG. 6 - An X-ray diffraction pattern obtained from a sample of fresh feed containing five per cent of a y type molecular sieve.

The (111) and (533) lines from sodium aluminium silicate hydrate are indicated on the chart (1×10^3 cps, TC=1, 1^o divergence slit, $1/2^o$ receiving slit, copper target).

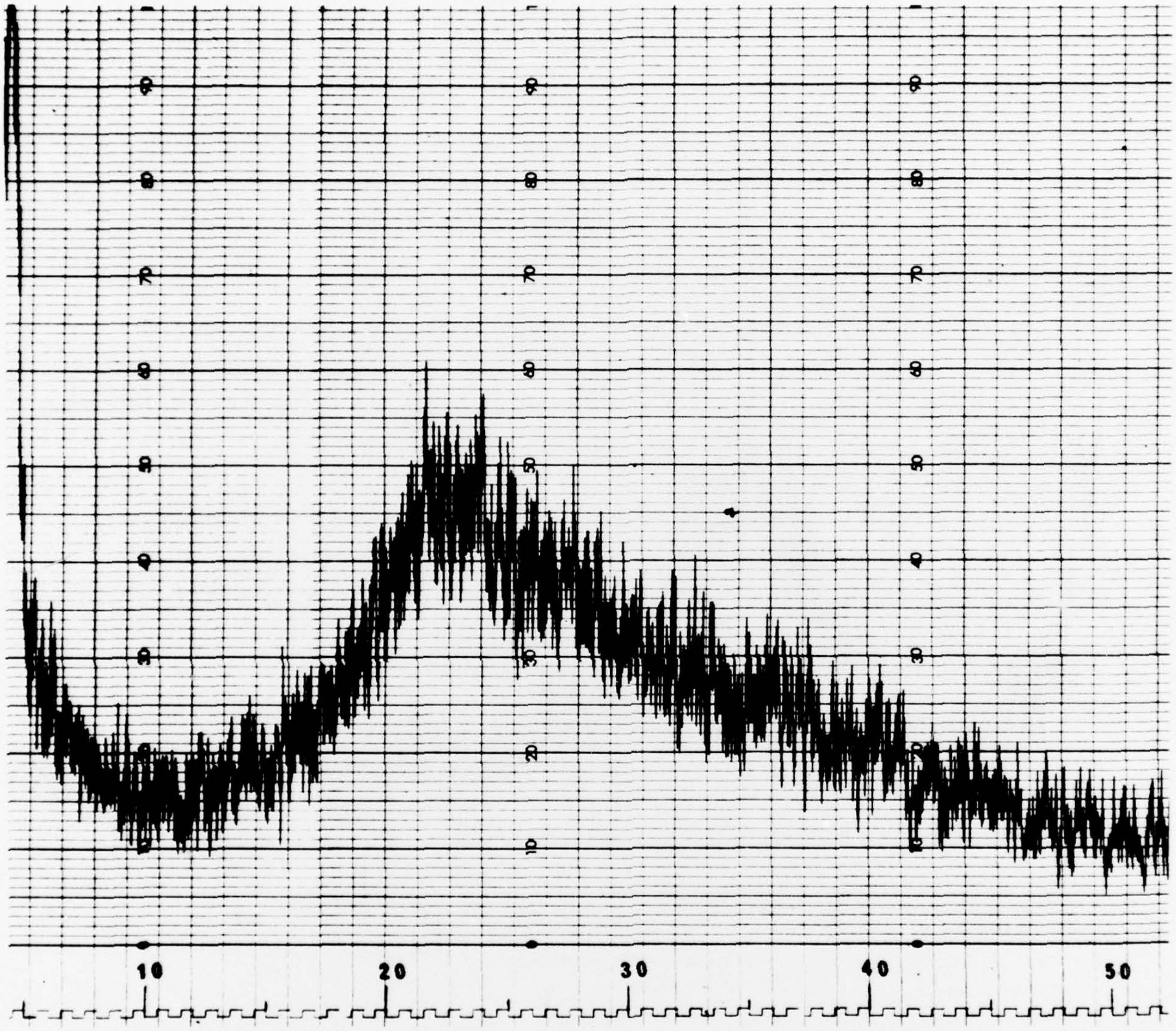

FIG. 7 - An X-ray diffraction pattern obtained from a sample of regenerated catalyst containing less than two per cent of "Y" molecular sieve (1×10^2 cps, TC=1, 1° divergence slit, $1/2^{\circ}$ receiving slit, copper target).

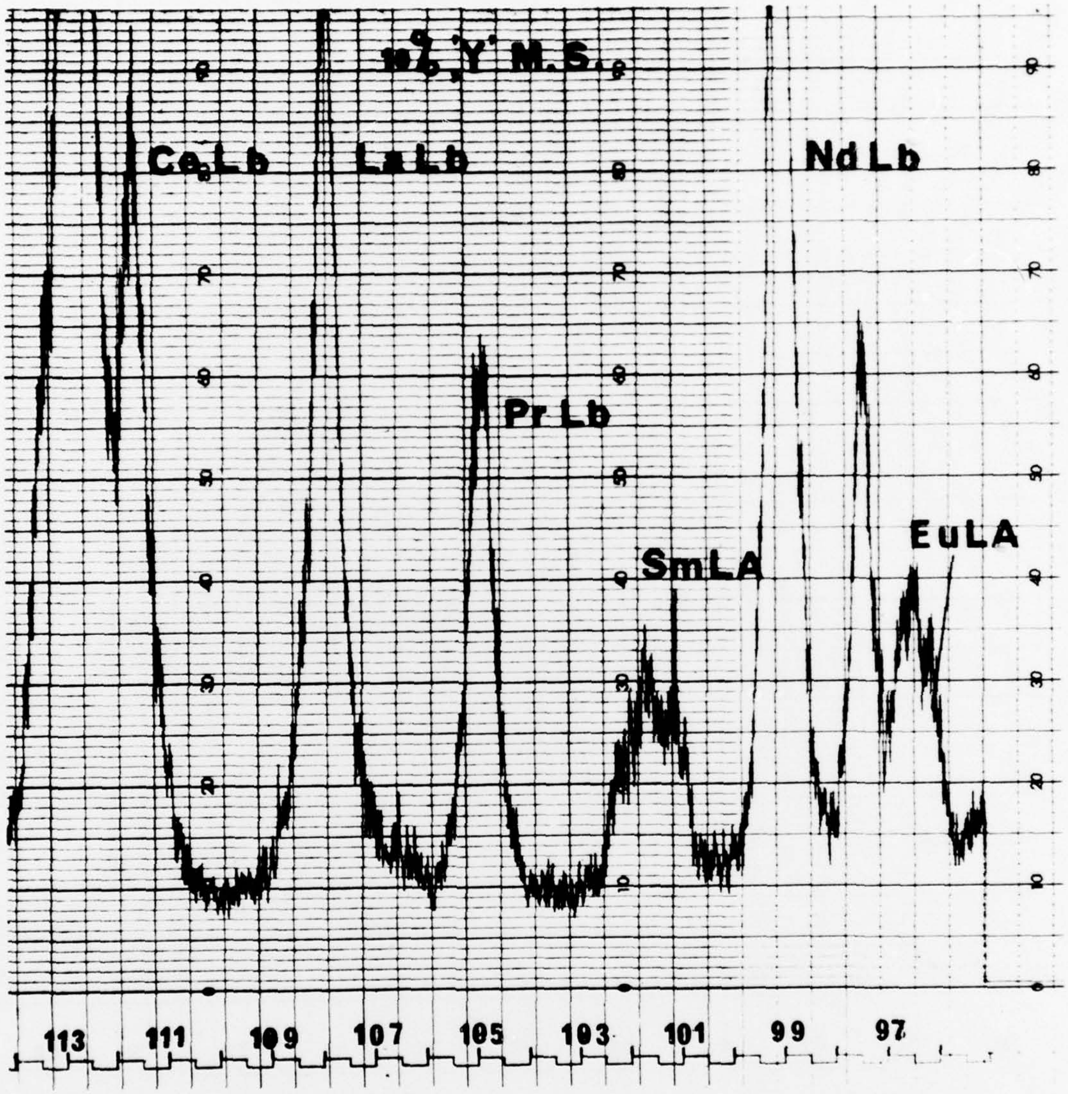

FIG. 8 - An X.R.F. scan of a silica-alumina catalyst containing 10% w/w "Y" type molecular sieve. (LiF 220 crystal, Range: 2 x 10^2 cps).

CORRECTION TO FIGURE 8. The following symbols should be changed from

Eu LA	to	Eu La
Sm LA	to	Sm La
La Lb	to	LA Lb
Nd Lb	to	ND Lb

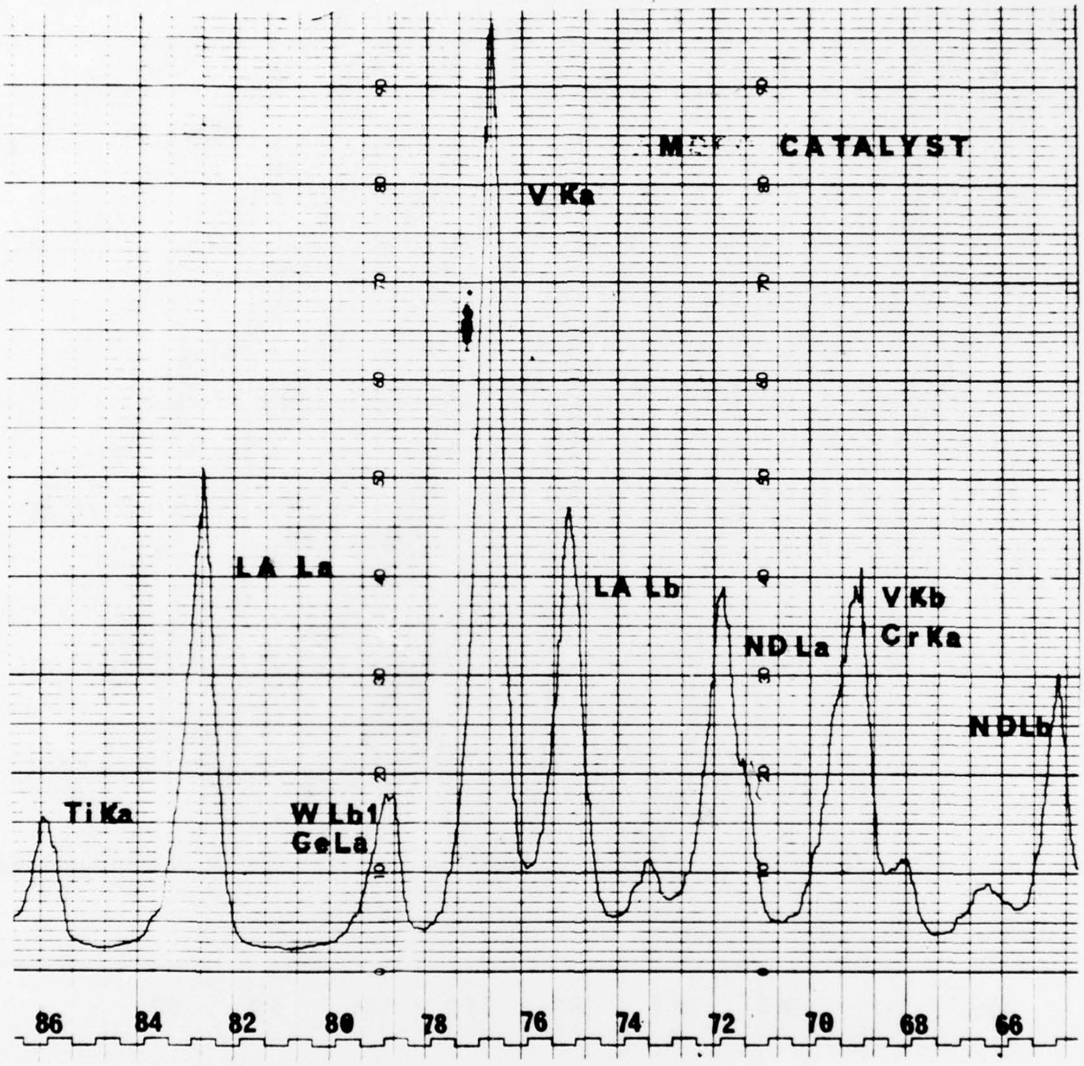

FIG. 9 - An X.R.F. scan of an "M" type catalyst. (Range: 1×10^3 cps).

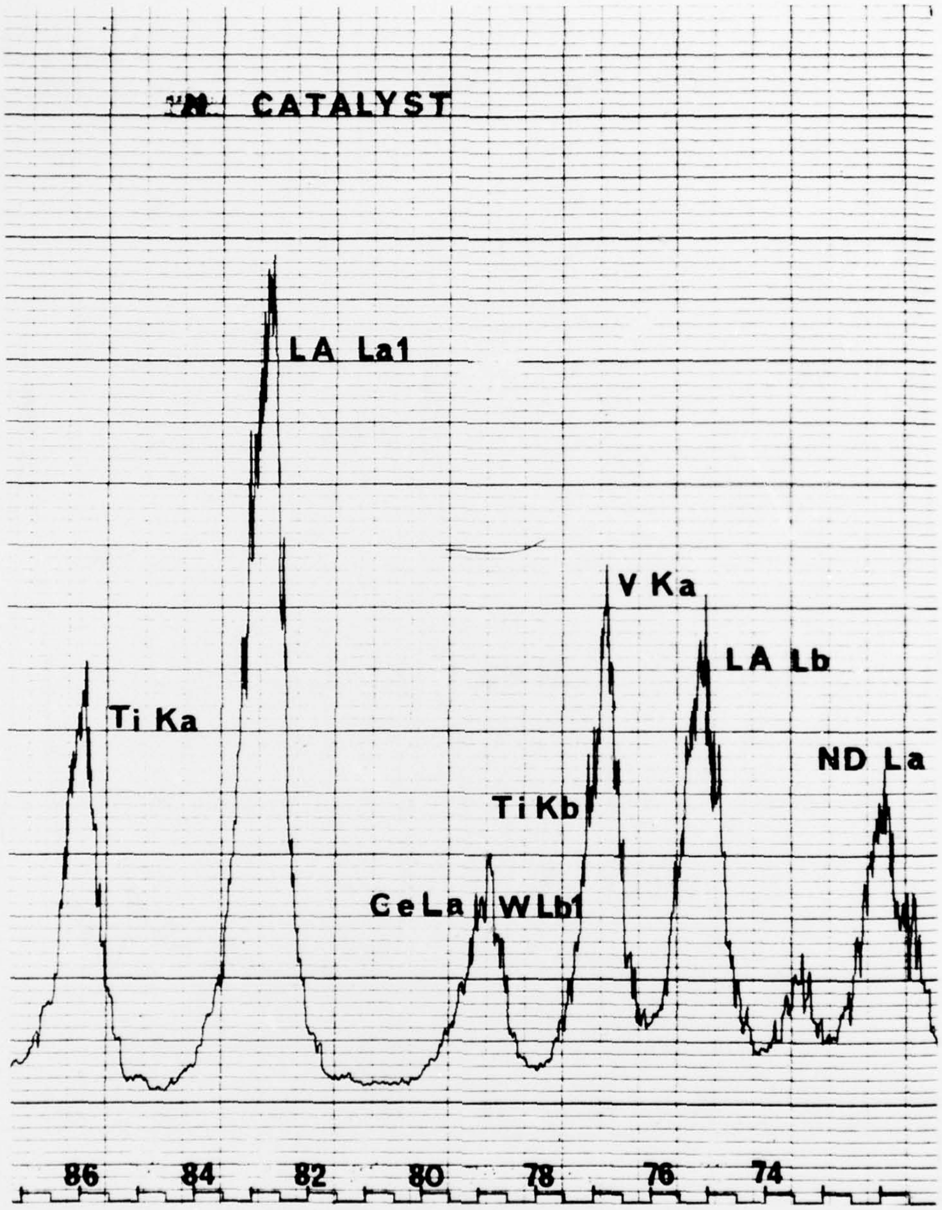

FIG. 10 - An X.R.F. scan of an "N" type catalyst.
(Range: 2×10^2 cps).

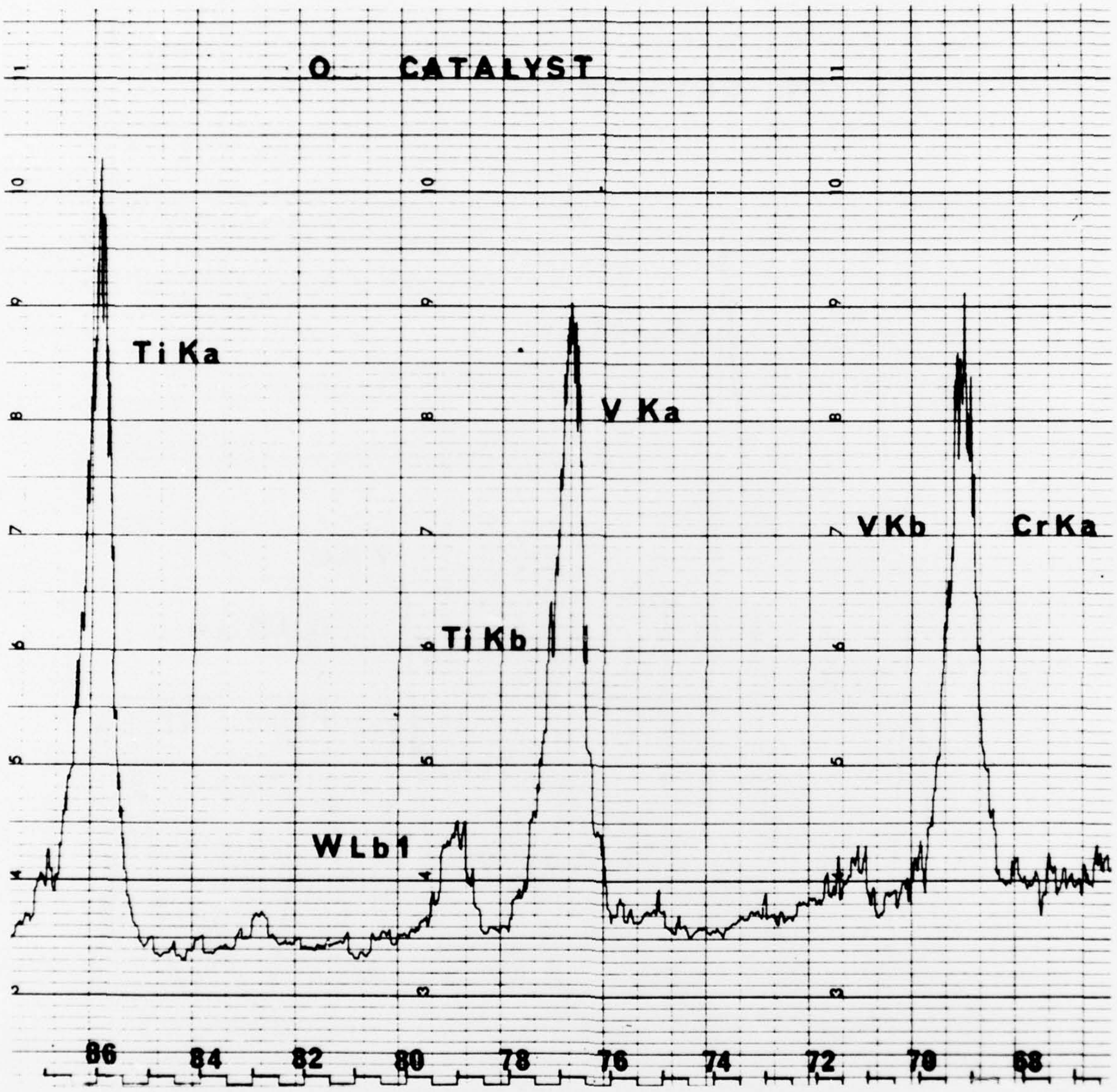

FIG. 11 - An X.F.R. scan of an "O" type catalyst. (Range: 2×10^2 cps).

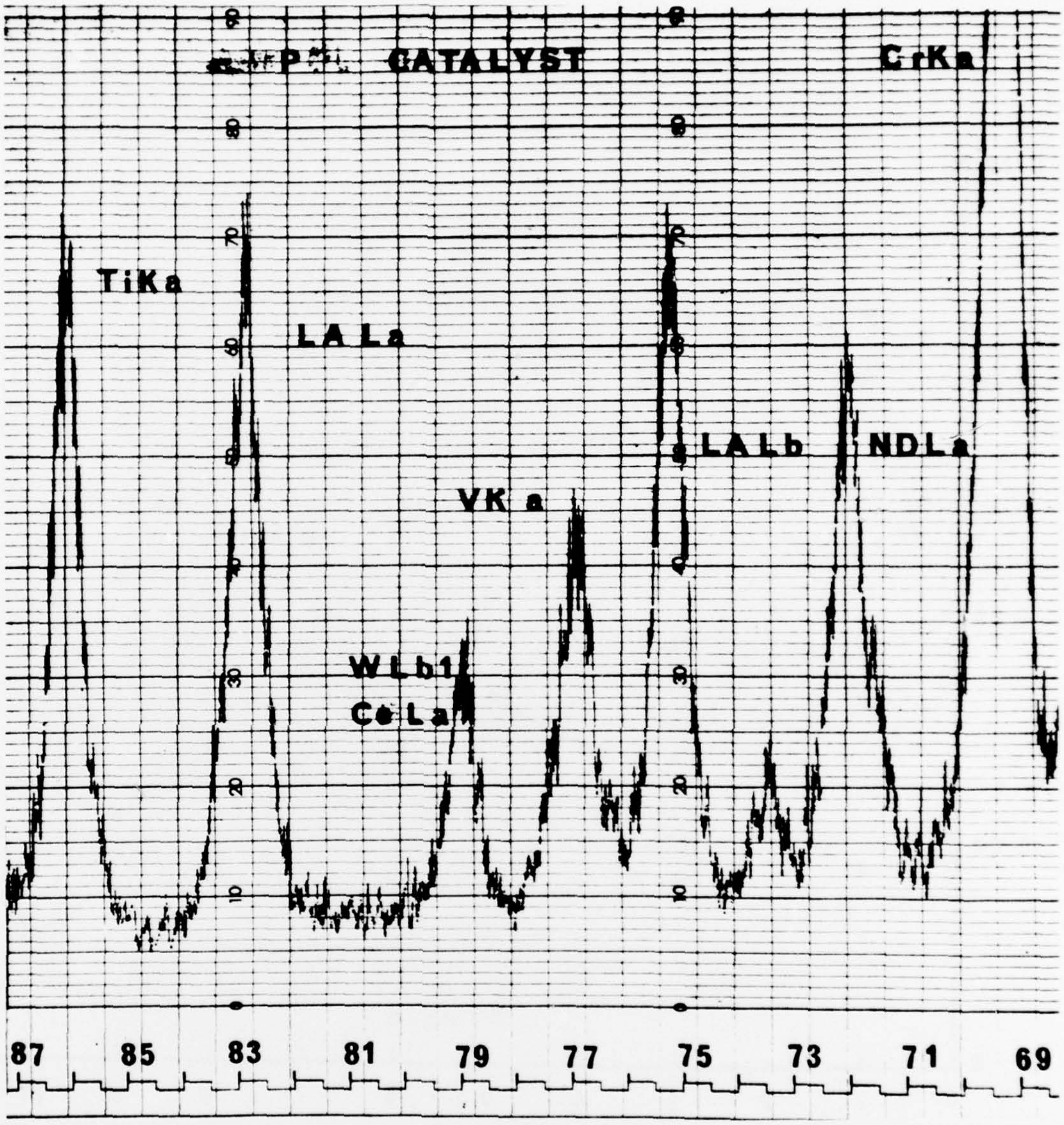

FIG. 12 - An X.R.F. scan of a "P" type catalyst.
(Range: 2×10^2 cps).

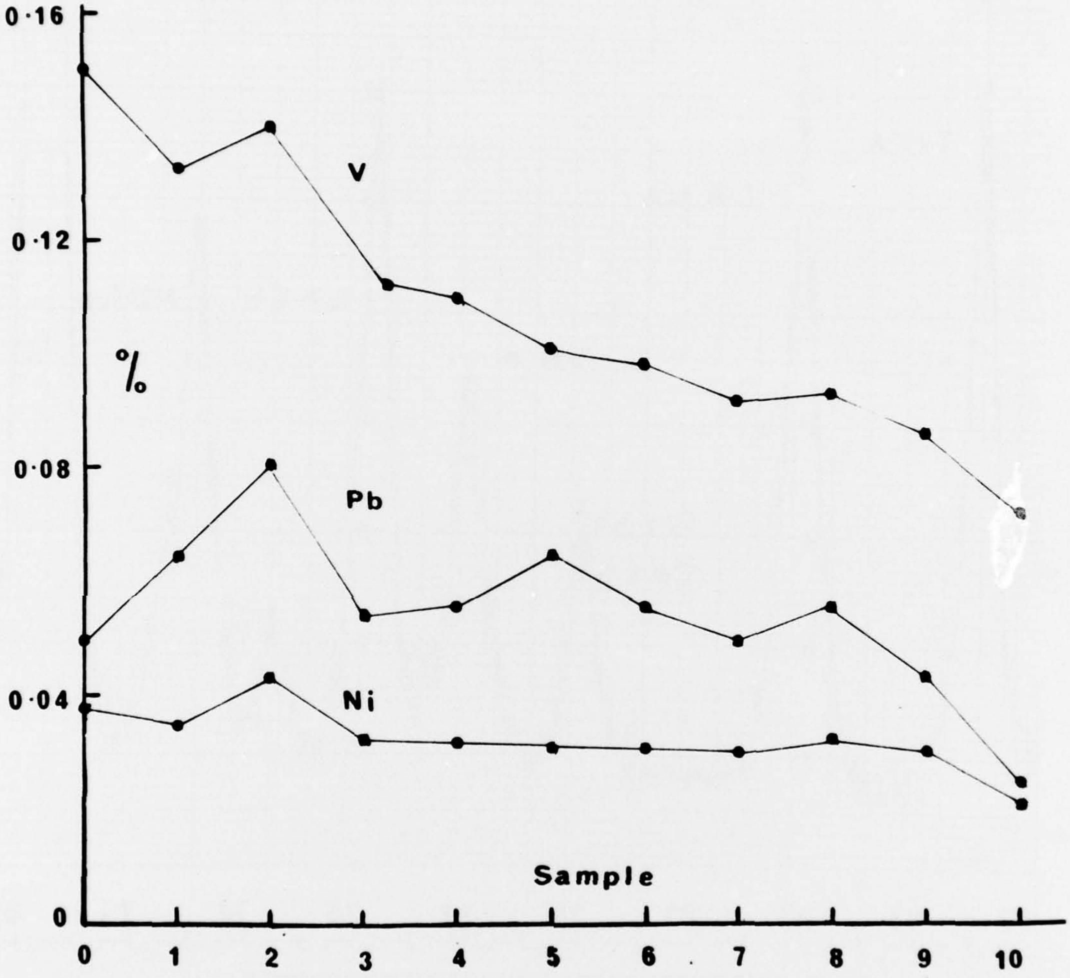

FIG. 13 - Variations in the concentrations of vanadium, lead and nickel in eleven consecutive samples of M regenerated catalyst.

APPENDIX

SYMBOLS EMPLOYED IN FIGS. 9-13

Elements

Ti	Titanium
V	Vanadium
Cr	Chromium
LA	Lanthanum
Ce	Cerium
Pr	Praseodymium
ND	Neodymium
Sm	Samarium
Eu	Europium
W	Tungsten
Pb	Lead
Ni	Nickel

Note: Lanthanum and Neodymium have been clearly distinguished by writing their symbols in capital letters.

Characteristic Emission Lines

Ka	K alpha line
Kb	K beta line
La	L alpha line
Lb	L beta line

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