The Recovery of Platinum from Reforming Catalysts

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The platinum-bearing catalysts now in such substantial use in the petroleum industry retain their high activity during many months of use, but ultimately they require to be replaced by fresh charges. The efficient and economic recovery of the platinum from spent catalyst is therefore of great importance to the commercial success of catalytic reforming. This article describes the methods adopted in the Johnson Matthey refineries, particularly for sampling, to achieve the highest possible recoveries of these residues.

The most notable development since the war in automotive engine design has been the steady increase in compression ratios, which has brought about a rising demand for high-octane motor spirit. This need has mainly been met by the introduction of platinum-catalysed reforming processes. Catalytic reforming can be applied to a range of naphtha fractions that are available in ample quantities from either American or Middle Eastern crudes, and gives an excellent yield of high-octane blending stocks that also have good lead susceptibility. The only other practicable method of octane-enhancement is by alkylation, and this process is less attractive economically and requires raw materials of more limited availability.

Catalyst Life

All platinum reforming processes are operated at relatively high temperatures and pressures, so that the activity of the catalyst mass gradually diminishes during use, and progressively increasing severity of operation is necessary to maintain the required octane level in the product. It has been found possible to prolong the active life of the catalyst, partly by pretreating the feed-stocks to remove sulphur compounds and other catalyst poisons, and partly by periodical regeneration of a part of the charge. Ultimately however the catalytic activity of the charge falls to an unacceptably low level, and it must be removed and replaced.

The catalyst charge is made up of small spheres or pellets of alumina or silica-alumina, impregnated with up to one per cent of platinum. The value of this platinum is a substantial part of the cost of the catalyst, so that ability to recover it almost completely from the spent mass is of essential importance to the economic success of the various reforming processes. It was clearly necessary, therefore, for a suitable process to be developed that would provide an efficient and economic method of recovery, and the design and development of large-scale plant for this purpose has in fact gone hand in hand with the manufacture of catalyst and erection of reformers.

Problems of Large Scale Recovery

There has always been a strong economic incentive to recover platinum metals after use, and refiners are accustomed to treating fairly large quantities returned either as metallic scrap or dispersed in sweepings and residues of various kinds. The large-scale recovery of platinum from a relatively refractory aluminous carrier is however a much more difficult problem, to which a number of different approaches are possible,
and the need to recover many thousands of ounces of platinum from spent reforming catalyst has initiated a vigorous search for the most effective method.

One of the most difficult aspects of the recovery problem is that of accurate sampling, and it is of course necessary to do this in order to make a precise determination of the platinum content of the spent mass, which will form the basis of payment. The material is returned for recovery in quantities of from 2,000 to 40,000 lb. weight, and the pellets are partly covered with carbon, impregnated with hydrocarbons, and accompanied by fines containing iron oxide and sulphide, and by miscellaneous debris. The overall platinum content will usually lie between 0.4 and 1 per cent, but it will vary slightly between individual pellets, and considerably between the pellets, the fines and the scale and oversize.

**Sampling Procedure**

A very elaborate sampling and assay procedure is therefore necessary to establish the true platinum content of returned material which is hygroscopic and must therefore be exposed to the atmosphere as little as possible. The sampling plant shown here was designed specifically for handling the substantial amounts of spent reforming catalyst that are now required to be recovered.

The material is sampled, if the amount is sufficient, in quantities of 4,000 lb. The contents of each drum are lifted in a skip hoist and discharged through a bottom-opening valve into a hopper over a screening machine. This is a totally-enclosed double-deck vibrator, with one 4 mesh and one 18 mesh screen, and in it the charge is separated into three fractions—oversize, fines and intermediate. The intermediates, containing all the whole pellets, go over a magnetic drum separator and thence to an eight-stage riffle which separates out a 1/256 part. Each 250 lb. drum of material is treated in this way, and the pellet samples are combined. A proportion is kept as pellets and the remainder is ground, coned, and split for assay. Ignition loss is determined on the unground pellets,

*The sampling plant in the Johnson Matthey refineries, specially designed for handling spent reforming catalyst*
and fines, oversize, magnetics and ground pellets are each treated separately and assayed for platinum content. In this way, a very accurate measure of the actual weight of platinum in the whole consignment is built up. Check weighings are made at the beginning and end of sampling to eliminate errors caused by changes in weight, and dust losses are carefully controlled.

After sampling, the spent catalyst is roasted under carefully controlled conditions to remove the organic contaminants and then goes forward for treatment. The operations are carried out entirely in closed systems from which no losses are possible, and all the effluents are monitored and treated, if necessary, for the recovery of traces of colloidal or particulate platinum.

Because of the accuracy of sampling and assay, and the control over all sources of platinum loss, that have been achieved in this plant, it is possible to carry out this difficult recovery operation at a very low cost and to make a major contribution to the overall economic success of platinum reforming in the petroleum industry.

Contamination of Electrical Contacts

A PLASTIC REPLICA METHOD OF EXAMINATION

Many millions of relays in the telephone and communications field depend for their successful operation on the contacts, and while the use of platinum and palladium virtually eliminates trouble due to tarnish films some inexplicable failures occasionally arise, more especially under very sensitive conditions. These are usually accounted for by the presence of dust, fumes, erosion products or other extraneous matter, but the examination of failed contacts and the identification of the contaminant is an extremely difficult and delicate task.

An elegant method of contact study has recently been described by two engineers of the Bell Telephone Laboratories, H. W. Hermance and T. F. Egan (A.I.E.E. Communications and Electronics, 1958, Jan., 756-762). The technique consists of pressing the warmed contact into a clear thermoplastic material, which reproduces faithfully all surface details in the contact in reverse relief, while solid deposits on the surface adhere to the plastic. Foreign material suspected of impairing contact operation is thus made available for microscopic study on a clear and relatively inert base to which chemical tests may be applied. Using this method, an astonishing variety of materials has been identified as causing contact failure in individual cases. Most open-circuit conditions examined were found to be due to fibrous dusts from workers' clothing, from packing materials or from insulating materials.

Two typical replicas of palladium relay contacts after failure in service. That on the left shows a large cotton fibre with smaller fibre fragments and some erosion dust. On the right, phenol fibre fragments are predominant.