Automated Procedures
for the Spectrophotometric Analysis of Steel and Slag

P. H. Scholes

The British Iron and Steel Research Association, Metallurgy Division,
Sheffield 3, Great Britain

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Summary. The Technicon AutoAnalyzer has been successfully used for the automatic determination of phosphorus, manganese, and silicon in most types of steel. Phosphorus and silicon are determined as molybdenum-blue complexes and manganese is determined as permanganate.
The system has also been applied to the determination of iron, manganese, phosphorus, and aluminium in steelmaking slags. For both steel and slag the sample is decomposed in acid, silicon removed as metasilicie acid and the solution diluted to a volume. From this stage the analysis is completely automatic.

Introduction
In the steel industry the main emphasis in analytical research is on physical methods such as direct reading spectrography and X-ray fluorescence, methods which provide a complete analysis in a few minutes. Thus in the future the job of the chemist will be to some extent subsidiary to the man operating electronic instruments. However it seems unlikely that the chemist will expire completely. The more difficult analytical tasks will still have to be solved by chemistry.
Most important the chemist must act as arbiter in cases of doubt. He must be prepared to analyse samples by reference methods with the highest possible precision and accuracy. Such tasks as these are often carried out by the research institute such as the British Iron and Steel Research Association or the central research laboratory in a large steel plant.
Thus in our Institute, in addition to purely physical techniques, we have attempted to assess and to utilize automatic and semi-automatic physicochemical instruments. Our intention being to replace conventional manual processes for high quality analysis. Automatic colorimetric analysis using the Technicon AutoAnalyzer is an example of this work.
The use of the AutoAnalyzer is well established in the clinical analysis of body fluids. It is becoming standard equipment in hospital laboratories both here in Europe and in the United States. In industry it has been used in many diverse fields ranging from the determination of streptomycin in fermentation to the determination of metals in soils obtained in geochemical surveys.
The principles of operation are as follows:
The sample if it is a solid such as steel must first be dissolved in acid and the solution diluted to a fixed volume. A small plastic cup, 2.5 x 1.0 cm in diameter, is filled with the sample solution; only a small volume is required and this need not be measured. The cup is placed on a turntable and from this stage analysis is entirely automatic. Solution is introduced into the apparatus through a plastic tube dipped into the cup. Aspiration is by means of a peristaltic pump operating by compressing the plastic tubing. After a fixed time interval the tube is removed from the cup and the turntable moves round to the next sample. Then the plastic tube dips into the second solution and so on. In this way either 20 or 40 samples per hour can be introduced into the apparatus.

Reagent solutions are continuously introduced into the apparatus. They are mixed with the sample solution in an arrangement of plastic tubes and mixing coils called a manifold. In the interval between samples, as the turntable moves round, dilute acid solution is introduced and this mixes with the reagents to form a barrier in the flow system between successive samples.

Air is also introduced and this splits the flowing system into small segments which are each separated by an air bubble. This effect is shown in Fig. 1. There are about 150 segments to each sample and introduction of air is a most important part of AutoAnalyzer technology. The bubbles act as a wiping medium on the plastic tubes and glass coils continuously sweeping residual matter from the walls into the preceding segment. The system is thus self-cleansing. The presence of air also promotes mixing between reagents and the sample during passage of the solution through the glass mixing coils.

At an appropriate stage, the colour forming agent is added and the coloured product is then passed through a colorimeter fitted with interference filters. The colorimeter monitors the optical density of the solution and its output is shown on a chart recorder in the form of a peak. The recorder is scaled directly in optical density units and thus peak heights may be related to a calibration graph prepared in the usual way.

We have used an AutoAnalyzer at our Institute for the analysis of certain components in steelmaking slag [1] and more recently we have used it to determine manganese, phosphorus, and silicon in steel [2]. I will describe its use in steel analysis first and then give a brief résumé of our earlier work on slag analysis.

Analysis of steel

A drawing of the instrument is shown in Fig. 2. This shows the unitised layout of the AutoAnalyzer: First, on the right-hand side of the Figure, the sampler unit which is a turntable holding plastic cups filled with test solution. Secondly, the peristaltic pump with its manifold of plastic tubes