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DETERMINATION OF IRON IN HIGH SIDERITE CONTAINING SAMPLES THROUGH WAVELENGTH DISPERSIVE X-RAY FLUORESCENCE SPECTROMETRY

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Abstract

A method is proposed for the analysis of Siderite bearing samples using Wave length DispersiveX-Ray Fluorescence Spectrophotometry (WD-XRF), with specimens being measured in the form of pressed pellets. Non-availability of Siderite certified reference material has been a limiting factor for carrying out analysis of siderite samples directly using WDXRF. Therefore the existing method of determining Total iron in hematite & magnetite samples has been modified to determine total iron in siderite samples.Preparation of synthetic standards containing $FeCO_3$, could be an alternative but, use of CRM/ SRM's have an advantage over synthetic standards, in terms of matrix matching with that of the specimen. The method presented in this paper will be helpful in case of siderite sample analysis especially whenWD-XRF is calibrated with hematite and magnetite CRM/SRM's. Initially, the proposed method involved calibration of WDXRF using ignited CRM/SRM's hematite, magnetite (NIST traceable) and In-house Iron ore standards followed by determination of total Fe as Fe_2O_3 in Iron ore specimen in the form of pressed pellet. The accuracy, precision and reproducibility of the method were also evaluated. Subsequently, determination of total Fe in siderite samples involved calcination/loss on ignition (LOI) followed by analysis by WD-XRF using PANalytical classical SuperQ regression model for matrix corrections. The method was validated through determination of total Fe and Fe^{2+} values in the samples using wet classical methods of analysis.

Keywords: Mineralogical Effect, Siderite, Calcination, Particle size effect, Hematite, CRM, WD-XRF