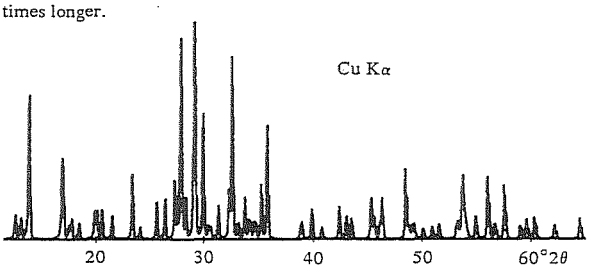
The method was tested with a large variety of organics, inorganics and minerals, in two to six compound mixtures using published examples and specimens run with our Series/1 automation system. Both peak search data reduction [Huang and Parrish, Adv X-Ray Anal. (1984) 27] and Search/Match were done on the host IBM 3083 computer. The figure of merit (FOM) was calculated from the weighted match of the d's, and the fractions of the standard and the unknown reflections matched in the experimental 2θ range. Chemical prescreening further reduced the search time. The correct compounds had the highest FOMs and were easily identified.

An example of the method is the analysis of the unknown pattern with 69 reflections shown below which was synthesized from published data [sample U-3.6 in Frevel, Anal. Chem. (1965) 37, 471]. Search/Match parameters were: error windows = 0.075°2θ, elements present = Y, As, Ag, Pb, Se and K; undetermined = H to F; and absent = remaining elements. Six candidates were found by Search/Match the frequently encountered phases (FEP) section of the 12 d's inorganics subfile: As_2O_3 (FOM=86), Ag_3AsO_4 (76), Y_2O_3 (75), $PbSeO_4$ (71), KH_2AsO_4 (69) and BeB_6 (41). The last phase (BeB_6) had a much lower FOM and was deleted. By comparing the matched table and plots, the true components in this mixture were easily identified to be the top five phases. Search/Match was done in 0.3 second CPU time. The CPU time for Search/Match of the FEP section of the regular inorganics subfile (standard patterns with all d's and I's) was more than three times longer.



12.2–2 QUANTITATIVE ANALYSIS OF THE POLYMORPHIC CONTENT OF CHLORAMPHENICOL PALMITATE BY X-RAY POWDER DIFFRACTION.
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Chloramphenicol palmitate (CMP) is known to exist in three polymorphic forms, designated as A, B and C (Aguiar, Krc, Kinkel and Samyn, 1967), or as β, α and γ, respectively (Szulzewsky, Kulpe, Schulz and Fichtner-Schmittler, 1982). The most stable form (A or β) is also the most resistant to enzymatic hydrolysis to chloramphenicol, and thus the least bioavailable. Although the polymorphic content of CMP is not controlled in the bulk drug substance, a compendial specification for CMP Oral Suspension requires that the content of polymorph A crystals be not more than 10 percent. The monograph in United States Pharmacopeia XX (1980) considers only "A" and "non-A" polymorphs, and quantitates the more desirable "non-A" polymorph by measurement of ratios of peaks in the IR spectra. The B and C polymorphs cannot be distinguished by their IR spectra.

Both the A and B polymorphs may be characterized by powder diffraction patterns which have the most intense peak at a very low angle (below 4° 2θ for CuKα X-rays). ZnO (rather than corundum) is used as an internal standard in the determination of a reference intensity ratio (RIR). This choice is based on reasons of convenience rather than technical reasons. ZnO has certain pharmaceutical uses; therefore, its purity is also subject to pharmacopeial standards and appropriate reference standard quality material is readily available.

When compared to the current IR absorption method, the use of ZnO to determine the RIR is found to be sufficiently sensitive to quantitate amounts of the undesired A polymorph in the 0-20 percent range. Preferred orientation effects, while significant for both polymorphs, are much less for the large peaks of the B polymorph. As a result, the precision of the quantitation is enhanced by an amount which exceeds the loss of precision due to preferred orientation. A procedure for quantitation of the polymorphic content of CMP, based on the above considerations, has been developed. Its validation for drug standardization purposes is described.

Aguiar, A. J., Krc, J. Jr, Kinkel, A. W. and Samyn, J. C. (1967). J. Pharm. Sci. 56, 847-853.

Szulzewsky, K., Kulpe, S., Schulz, B. and Fichtner-Schmittler, H. (1982). Acta Pharm. Suec. 19, 457-470.

USP XX. (1980). United States Pharmacopeial Convention, 12601 Twinbrook Parkway, Rockville, Md. 20852.

12.3–1 X-RAY POWDER DIFFRACTION STUDY OF RUBIES.
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Ruby is the red gemstone in the corundum species. As with all gems, the value of corundum gems depends on color, clarity, and the quality of cutting. In general corundum crystallizes in the hexagonal system, the chemical compound is aluminum oxide (Al_2O_3), and pure corundum is colorless. But gems in this specie have different colors caused by the impurity. Ruby has different colors such as red, purple-red and medium to dark red that depend on the quantity of chromic oxide (Cr_2O_3) in the aluminum oxide compound. Variety of colors of natural ruby and synthetic ruby are compared by powder photographs taken with a Guinier-Hagg focusing camera. The powder diffraction data are obtained in the form of tables. The unit cell dimensions of different rubies are refined by the least-squares method. The ratio of impurity in Al_2O_3 to different color of ruby is identified by the powder diffraction method.