TECHNICAL NOTE

Barry K. Logan,[1] *Ph.D.; H. Steve Nichols,*[1] *B.S.; and David T. Stafford,*[1] *Ph.D.*

A Simple Laboratory Test for the Determination of the Chemical Form of Cocaine

REFERENCE: Logan, B. K., Nichols, H. S., and Stafford, D. T., "**A Simple Laboratory Test for the Determination of the Chemical Form of Cocaine,**" *Journal of Forensic Sciences,* JFSCA, Vol. 34, No. 3, May 1989, pp. 678-681.

ABSTRACT: The authors describe a simple chemical test to determine whether a suspect specimen contains cocaine and if so whether it is present as free base cocaine or in a salt form. The distinction is based on the relative solubility of the two compounds in aqueous or organic solvent. The test, based on the widely accepted cobalt thiocyanate color reaction, is performed in three stages to make the interpretation as complete as possible.

KEYWORDS: toxicology, cocaine, drug identification

A thorough analysis of cocaine is currently an involved process, requiring unequivocal identification of the chemical structure and also, under some states laws, the stereochemistry of the molecule [*1*]. A quantitation is also normally included. Changing trends in cocaine abuse, however, have added the requirement for a further determination, namely that of the chemical form of cocaine as either free base or salt.

Cocaine hydrochloride is the usual end product of the cocaine refining process [*2*], and this is normally taken by insufflation. Recently, however, the smoking of free base cocaine or "crack" has become popular among cocaine users as it gives a sudden intense high and administration can be repeated at regular (10-min) intervals without any diminution in effect. Smoking free base cocaine is generally held to be "more dangerous" because the drug is absorbed rapidly, resulting in very high blood concentrations which can cause death through heart or respiratory failure or both.

In practice, the distinction between the free base and the salt forms is rather academic, as the interconversion of one to the other can be achieved in a few minutes using household chemicals [*2,3*]. Under current federal law, however, the possession of greater than 5 g of free base cocaine carries an equivalent sentence to possession of 500 g of the salt [*4*].

The analytical determination of the chemical form of cocaine is therefore important from a forensic science point of view. Various methods for the determination of the chemical form

Received for publication 17 May 1988; revised manuscript received 29 July 1988; accepted for publication 4 Aug. 1988.

[1]Research toxicologist, laboratory supervisor, and laboratory director, respectively, Toxicology Laboratory, Department of Pathology, University of Tennessee, Center for the Health Sciences, Memphis, TN.

of cocaine have been considered. The salt and free base forms of cocaine can be differentiated on the basis of their infrared (IR) spectra [5]; the possible presence of other drugs or nondrug material in the specimen, however, makes this procedure more difficult. The use of silver nitrate ($AgNO_3$) to test for the chloride counter ion from the salt has been considered; however, some of the processes used for the preparation of free base cocaine [2,3] may result in free base that contains chloride ions both from the original salt and as a contaminant in domestic grade sodium bicarbonate. An additional problem of this test is its lack of specificity, as $AgNO_3$ shows a marked reaction with bicarbonate ion—a common contaminant of free base cocaine [2].

Even specific tests for the presence of counter ions, such as ion chromatography, are of limited use, as they require the dissolution of the specimen in an aqueous phase, a process which may change the chemical form of the cocaine from its original state. For example, free base cocaine containing sodium chloride would give the same result as pure cocaine hydrochloride.

Similarly, dissolution in water of a mixture of cocaine hydrochloride and sodium bicarbonate (a possible precursory mixture in free base cocaine preparation) would complete the reaction changing the chemical form of the cocaine from its original state.

Summerhays has described a method involving a hexane or pentane extraction followed by gas chromatographic (GC) analysis to determine the presence of free base in a cocaine specimen [6]. This method is however susceptible to misinterpretation in the last example cited above, and requires at least one additional GC analysis, increasing demand on instrumentation and reducing sample throughput. It is also limited to laboratory use.

The solubility properties of the free base and salt forms do however provide the best method for differentiation, and this principle was used in the development of the test described below, which is based on the cobalt thiocyanate color test already in general use [7].

Method

A specimen suspected of containing cocaine is triturated, and 5 to 10 mg are added to Test Tube 1 containing 1 mL of hexane. The tube is shaken vigorously or vortexed for about a minute to allow any dissolution of the sample to take place. The sample is centrifuged or allowed to stand to remove any suspended particulates.

Test 1

A six- to ten-drop aliquot of the hexane is transfered to a second tube and four to six drops of cobalt thiocyanate [$Co(SCN)_2$] solution [8] is added and shaken briefly. The appearance of a characteristic blue precipitate indicates the presence of free base cocaine in the sample.

Test 2

One millilitre of water is added to the remaining hexane and any undissolved sample in the original tube. This is shaken briefly to allow any dissolution of the sample to take place. A four- to six-drop aliquot of the aqueous layer is transfered to a test tube containing 0.5 mL of chloroform. This is shaken briefly and four to six drops of cobalt thiocyanate solution is added. The appearance of a blue color or precipitate in the chloroform layer indicates the presence of a cocaine salt in the sample.

Test 3

In the case of a negative result in both Tests 1 and 2, the remaining hexane in Tube 1 is sampled and six to ten drops are retested with four to six drops of cobalt thiocyanate solu-

TABLE 1—*Interpretation of test results.*

Test 1	Test 2	Test 3	Cocaine Present?	Conclusion
+	+		Y	Free base and salt present.
+	−		Y	Free base present. No salt.
−	+		Y	No free base. Salt present.
−	−		?	GO TO TEST 3.
		+	Y	Salt present together with sodium bicarbonate ($NaHCO_3$).
		−	N	No cocaine present

tion. A positive result indicates that the initial sample is a mixture of cocaine hydrochloride and sodium bicarbonate, a possibility mentioned in the introduction. A negative result confirms the absence of any cocaine in the sample.

Results and Discussion

Table 1 shows the combinations of possible results for the three tests and the interpretation of these results. The basis of the test is the differential solubility of the free base and salt forms of cocaine in hexane. The presence of cocaine is indicated by the formation of the characteristic blue cocaine/cobalt complex.

The use of this three-level test is necessary to identify all the possible combinations discussed earlier. In its simplest form however, Test 1 alone, it can be used to screen for the presence of any free base.

Test 3 is necessary to identify the possibility of a cocaine hydrochloride/sodium bicarbonate mixture. To date, several specimens examined routinely in this laboratory have proven positive in this test.

The identification of cocaine by this method is only as specific as the cobalt thiocyanate test itself. This test is however a widely accepted presumptive test for cocaine; its limits are well understood, the subsequent confirmatory analysis is, of course, required [*9*]. The confirmation may be performed directly on the remaining hexane from the initial test tube. Confirmatory analysis on the aqueous fraction can be done by the extraction of the aqueous layer with chloroform and direct analysis of this by gas chromatography/mass spectroscopy (GC/MS).

The procedure described above has been used with considerable success in screening suspect specimens for the presence of free base cocaine. Run in batches of twelve, the procedure as described takes 15-20 min. To date, most of the specimens examined by this method have been either free base or salt form, although mixtures of the two have also been identified. This could result either from incomplete conversion during free base preparation or possibly from a specimen of the crude intermediate paste produced in the initial extraction of cocaine from the coca leaf [*2*].

This test is currently somewhat involved for use in the field, but a suitable packaged form is in development.

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Address requests for reprints or additional information to
Barry K. Logan
University of Tennessee
Toxicology Laboratory
3 N. Dunlap St.
Memphis, TN 38163