Demo of Chemically-Specific Non-Intrusive Detection of Cocaine Simulant by Fast Neutron Atometry

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ABSTRACT

We report a major advance in Fast Neutron Activation with Associated particle Production that opened up a novel inspection technology, named atometry; it procures, non-intrusively, remotely and on-line, the quantitative empirical chemical formula of inaccessible substances imbedded in metal or other materials. In 'Atometer 1,' we improved gamma energy and timing resolutions by a factor of 30 (from 6 to 0.2 %) and 15 (from 90 to 6 ns) respectively, by introducing Ge detectors and novel electronics. Analysis of entire gamma spectrum is obviated by digitizing selected lines - a gain of 10x. An algorithm based on Dalitz Triangle was devised that gets on-line substance recognition and chemical imaging. We retrieved, through ¾ inch of steel, the formula of non-hydrogen part of explosive simulant to be $C_{1.08} N_{1.95} O_1$ vs. true formula CN_2O. Next, we used Atometer 1 to get chemical formula of 10 lb of cocaine simulant in air and in rice. For non-hydrogen part of cocaine simulant it is: $C_{14}N_1O_{3.17}$. Our result in air is to within 1 st. dev. accurate: $C_{(14.6\pm.0.60)} N_{(1\pm 0.19)} O_{(3.28\pm 0.10)}$. Similar result obtained through rice will be also presented. A state-of-art atometer using commercial miniature neutron source producing 2×10^8 n/s/4p, and electronics parallel processor will chemically recognize 500 grams of substance in 60 sec or 5Kg in 6 sec. Radiation hazard will be less or equal to X-ray detector.

1. Objective of the Test.

Objective of the test was to determine whether or not a remote on-line chemically-specific recognition of cocaine simulant in open air and buried in rice can be made by the new detection technique fast neutron *atometry*. An experimental system named Atometer 1 at DOE's Special Technologies Laboratory in Santa Barbara, California, was used in the Test. Atometer 1 is an improvised system based on an old Fast Neutron Analysis device, built in 1984; which operated at a speed 40-80 times slower than Atometers with state-of-the-art components.

The complete empirical formula of the cocaine simulant supplied by Applied Technology Division of US Customs (normalized to nitrogen=1) is compared with the real cocaine formula below:

Cocaine simulant : $\qquad H_{15}C_{14}N_1O_{3.16}Cl_{0.67}$…………………………………………….(1)

Cocaine : $\qquad H_{22}C_{21}N_1O_4Cl_1$…………………………………………..(2)

2. Atometry

The technique of atometry was developed by the vendor in 1997-8, in collaboration with two laboratories of US DOE. Atometry represents a major advance in Fast Neutron Analysis with Associated Particle Imaging ('FNA/API') that opened up a novel drug inspection technology. An atometer obtains, non-intrusively, remotely and on-line, the quantitative empirical chemical formula of inaccessible substances imbedded in metal or other materials. In 'Atometer 1,' we improved gamma energy and timing resolutions by a factor of 30 (from 6 to 0.2 %) and 15 (from 90 to 6 ns) respectively, by introducing Ge detectors and novel electronics. Analysis of entire gamma spectrum is obviated by digitizing selected lines - a gain by a factor of 10. An entirely novel data reduction and analysis technique based on Dalitz Triangle was devised for on-line substance recognition and chemical imaging.

Unlike the early FNA techniques – which can give only a qualitative indication of a possible illicit substance with a very large false alarm rate - Atometer I has demonstrated its ability to unambiguously recognize, through 3/4" steel walls, the non-hydrogen part of quantitative empirical chemical formula of an explosive or drug in the form

$$C_{C'}N_{N'}O_{O'}\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots(3)$$

where C', N' and O' are relative atomic proportions of carbon, nitrogen and oxygen.
Every substance is represented by a dot in the 'atometric triangle' whose coordinates are $C_2N'_2O'_2$. E.g. for urea, as explosive simulant, we obtained the following formula :

$$C_{1.08}N_{1.95}O_1\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots(4)$$

which is to within $(+0.08-0.05):4=0.75\%$ equal to the correct formula, CN_2O

3. Results I: Chemically specific recognition of cocaine simulant in fast neutron atometric triangle.

In the first test, we used Atometer 1 to get chemical formula of 10 lbs. of cocaine simulant in air. The spectrum of cocaine hydrochloride obtained by HiEnergy is shown in Figure 1 in comparison with that obtained by Argonne National Laboratory [Dickerman, Rhodes, *Associated-Particle Sealed-Tube Neutron Probe: Detection of Explosives, Contraband, and Nuclear Matrials*, 1996]. After the standard atometric analysis has been applied to the data, the C',N' and O' values obtained from the atometric measurements are shown in Table 1. The result is that, for 3-element atometry data yield the formula that is to within 2% equal to the formula of the cocaine simulant.

For non-hydrogen part of cocaine simulant is:

$$C_{14}N_1O_{3.17}\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots(5)$$

Our result in air is to within 1 st. dev. equal to it:

$$C_{14.6\,(\pm0.60)}N_{1\,(\pm0.19)}O_{3.17\,(\pm0.10)}\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots(6)$$

Table 1.
Empirical Chemical formula of Cocaine Simulant

Run	Carbon	Nitrogen	Oxygen
128	16.3±0.45	1±0.25	3.22±0.24
138	14.6±0.60	1±0.19	3.28±0.10
Simulant	14	1	3.17
Cocaine	14	0.67	2.67

3.1 Speed of chemically-specific detection

From our measurements we extrapolate that a state-of-art atometer using commercial miniature neutron source producing 2×10^8 n/s/4p, and an electronics parallel processor operating in 10 nanosecond per event range, will chemically recognize 500 grams of substance in 60 sec or 5Kg in 6 sec. With 2×10^9 n/s/4p, the times will be reduced to 6 and 0.6 sec, respectively.

3.2 Radiation hazard

Radiation hazard with 2×10^8 n/s/4p, will be less than or equal to that from an X ray detector. We point out that by the use of the cocaine "primer", the atometer will operate only 10% of the time, thus a neutron flux of 2×10^9 will be equivalent to the radiation hazard from 2×10^8.

4. Limitations of the Test.

There are two limitations to the test:

1.2.1 Slowness. Atometer 1 is an imporvised system consisting of old electronics and other components that cannot handle the intense particle sources required to make atometry practical. It operates at a 40-100 times lower event rate (slower detection speed) than a system that would incorporate various innovations in the field and state-of-the-art components. Hence, the detection time of the described test should be divided by a factor of 100 to get an idea of performance of the Atometer that would be manufactured for the US Customs Service.

1.2.2 Single Voxel imaging or *'Bulk Imaging'*. Atometer 1 had its imaging detection system turned off in the tests, to that the matrix of 1,000 alpha scintillation counters acted as a single detector. Instead of getting the atometric plot for each of 1,000 voxels of the order of cubic centimeters each, the atometer viewed one large voxel of 200 liters volume. As a result, the atometric data reported below are averaged over many voxels, weakened by an order of magnitude for each order of magnitude of the averaging hence diluted. The sensitivity (speed) of chemical recognition of drugs has been weakened at the same rate. Therefore, the reported results can be considered as a "tip of the iceberg" as to the capacity of atometry.

5. Results II: Chemically specific recognition of cocaine simulant buried in rice with fast neutron atometry.

There were 4 measurements, referred to as the Runs:
Run A: 100% rice
Run B: 75% rice, 25% cocaine
Run C: 67% rice, 33% cocaine
Run D: 100% cocaine.

Applying the standard atometric analysis, we obtained the empirical chemical formula for the Run A (100% rice) to be:
$$C_{1.71} N_1 O_{4.15} \quad \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (7)$$
It is remarkable that, for Run C (67% rice, 33% cocaine), after background subtraction we get:
$$C_{14.3} N_1 O_{3.16} \quad \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (8)$$

which is to within 97% equal to the formula of the cocaine simulant, Eq. (1). Atometric Dalitz Plot is shown in Fig. 2 -- note that the theoretical and experimental points for pure cocaine (Run

D) overlap to the point that they are indistinguishable. For Run B (75% rice), we obtained an inaccurate formula (see Table 2). An impromptu interpretation of this would be that 3-element FN atometry cannot be used for the cargo/cocaine mass packing ratio from all sides greater than 2 to 1 per voxel. More studies are needed to determine if this limit can be exceeded.

Table 2.
Empirical Chemical formula of Cocaine Simulant in Rice

Run	Carbon	Nitrogen	Oxygen
A -- 100% Rice	1.71±0.29	1±0.15	4.15±0.14
B -- 75% R + 25% Coc.	14.3±0.52	1±0.19	0±0.23
C -- 67% R + 33% Coc.	14.3±0.48	1±0.22	3.16±0.12
D -- 100% Cocaine Sim.	14.6±0.32	1±0.08	3.17±0.19

5.1. Technical performance
The *time-of-flight spectra* in Runs B and D are shown in Figure 3. It is evident that the FWHM of the resolution curve is 22 channels, 0.29 ns each i.e. **the time resolution** of our alpha-gamma coincidence circuit was **6.38 ns** or **32.5 cm** in the z direction of the neutron flight path.
Signal-to-background ratio. The directly observable tagged neutron gamma spectrum ('raw spectrum') for Run B is shown in Fig. 5.. The spectra for Run B are shown in Fig. 6. The observed signal-to-noise ratio (peak-to-background) is 11±4. This is about 55 times that obtained in the previous Fast Neutron studies, which had a typical signal to noise ratio of 0.2±.05. [Vurvoupoulos. et al, *Proc. Counterdrug Law Enforcement Symposium* 2-4 (1995)].

5.2. Result 2: Atomic C/O ratio *versus* rice/cocaine mass ratio.
Prior to the advent of atometry, the atomic carbon -to-oxygen ratio obtained from the gamma line intensity, C/O, was used as the drug indicator. For cocaine and rice, C/O= 5 and 1, respectively. Omnipresence of the oxygen background renders the observed C/O always much lower. W.K.U. observed C/O=0.636 for pure rice and 0.701 for 70% rice, 30% cocaine, i.e. an 11% increase which is not sufficient for a prompt practical drug detection.

Our observed C/O values are:
Run   A   B   C   D
C/O   0.41   0.60   0.61   4.6 …………………………………………………………………..(9)
It is evident that, using Atometry, C/O increases between A and B by 50%.

5.3. Result 3: Cocaine-in-Cargo detection time via fast ratio of C/O.
The 50% increase makes the FN C/O ratio a practical instant indicator of, or "alert signal" for possible drug presence in a cargo. A 50% effect can be observed with a 10% statistics i. e. 200 events per gamma peak which would detect a 50% difference with 4 standard deviations e.g.: 240-160=80±20. The time required to obtain this statistics with a state-of-the-art Atometer 2 is $T_{C/O}$ @ 10 sec. …………………………………………………………………………………(10)

5. Experimental Setup and Procedure
A diagram of the experimental setup is depicted in Figure 6. The STL system has one position sensitive photomultiplier that uses charge division to give an analog position signal (10_4 elements), which is then digitized into an ADC.
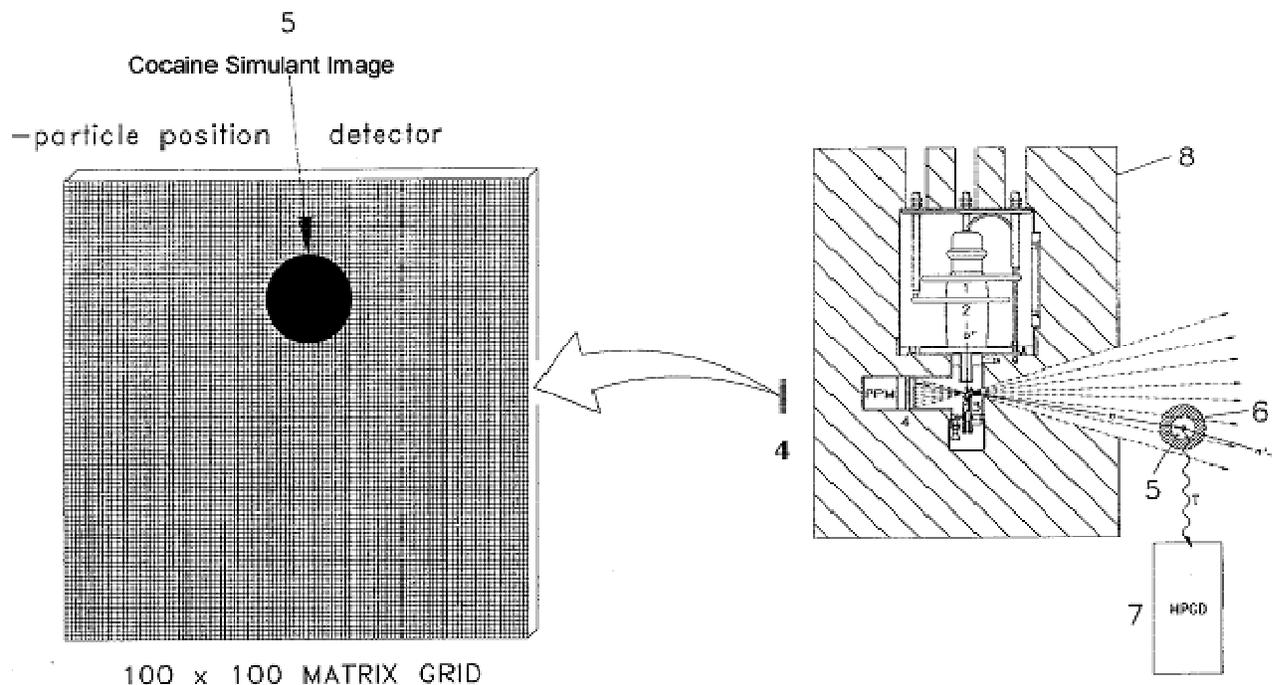
Fig. 6. Atometer 1. Setup for CHEMICAL RECOGNITION & IMAGING by Continuos beam Fast Neutron Analysis/ Alpha Particle Imaging. Beams of D_2 + ions (2) from mini accelerator (1) is incident onto scandium tritide target (3) at which n's and a particles are produced at 180_0 to one another. The a (emitted to the left) is detected by a fast (1.4 ns) phosphor deposited on the inner surface of a fiber-optic window (4) viewed from the outside by a position sensitive photomultiplier (PPM), with a space resolution of 0.8 mm FW in both x and y. The n (emitted to the right) passes through 3/4" steel shell undisturbed and undergoes resonant inelastic scattering with a nucleus of C or N or O of the Drug; (5) Kinetic energy difference of neutron before (n) and after (n') scattering is carried by a g ray which is detected in a high resolution (0.1%) germanium crystal (6) - but only if it is produced in coincidence with the a. (7) 80% N-type HPGe manufactured by EG&G Ortec; (8) Heavy polyethylene shield.

The cocaine simulant target (mass=10 lb., dimensions 10 cm X 28 cm X 28 cm) is positioned 109 cm from the neutron source. This source (a D -T neutron generator, with an adjustable strength of $5x10_5$ to $2x10_7$ neutrons/sec into 4p) is surrounded by a high density polyethylene collimator-shield. Positioned 10 cm behind the neutron source is a detector of the recoil a 's that are produced in coincidence with the neutrons that illuminate the cocaine simulant target. This allows us to "tag" each neutron that leaves the source to impinge on the target with the time at which it was generated, and in future experiments the angle at which it was emitted (API imaging mode). An overview of the experimental setup and the detection system electronics used in these tests is shown in Fig. 7.

The g-rays produced by the interactions of the neutrons with the simulant target are detected by a 80% efficient (Relative NaI), N-type EG&G Ortec HPGe detector positioned 35 cm from the center of the target. This detector is partially shielded from unwanted g-rays (for example those produced in the moderator-shield) by a 10 cm thick lead brick shield placed between the HPGe detector and the polyethylene neutron source shield.

The electronic system used at present produces 3 types of spectra that provide information about

the target being examined:

1): The ungated g-ray spectrum which is the number of g 's vs. energy for all g-rays detected by the HPGe detector. This is accomplished by simply monitoring the output of the detector (after amplification by a spectroscopy amp) by a multichannel analyzer- computer system (Ortec DART and a Pentium class PC) with no regard for the timing information produced by a detector. This is also called the TNA spectrum because the lack of time correlation permits the detection of g-rays produced by thermal neutron activation. Since the neutrons involved in these reactions require a substantial amount of time to slow from their original 14 MeV energy to only several eV, all timing correlation with their original production time is lost, and they would not be recorded if time gating was used.

2): The gated g-ray spectrum which is the spectrum produced only by those neutrons whose arrival time at the target zone is consistent with their moment of production in the neutron source (i.e. only "tagged" neutrons) within a small time window. For these experiments this time window was 6 nanoseconds. Since 14 MeV neutrons have a velocity of 5 cm/nanosec this corresponds to a zone within +/- 15 cm from the center of the target when this window is properly adjusted (i.e. when the affect of neutron time of flight from the source to the target and intrinsic electronic time delays are taken into account). This spectrum is also known as the FNA spectrum since it can only be caused by the 14 MeV neutrons produced with the proper time correlation with their production at the neutron source. This is accomplished in the electronics by passing the outputs of both the g and a detectors through separate fast amplifierdiscriminator
circuits which change the outputs of the detectors into sharp time-accurate timing pulses (albeit with the loss of the amplitude-energy information). These pulses are then passed through adjustable electronic time delays to compensate for neutron and g time-of-flights, etc. and sent to a timing coincidence circuit. If these signals both arrive within a specified time.
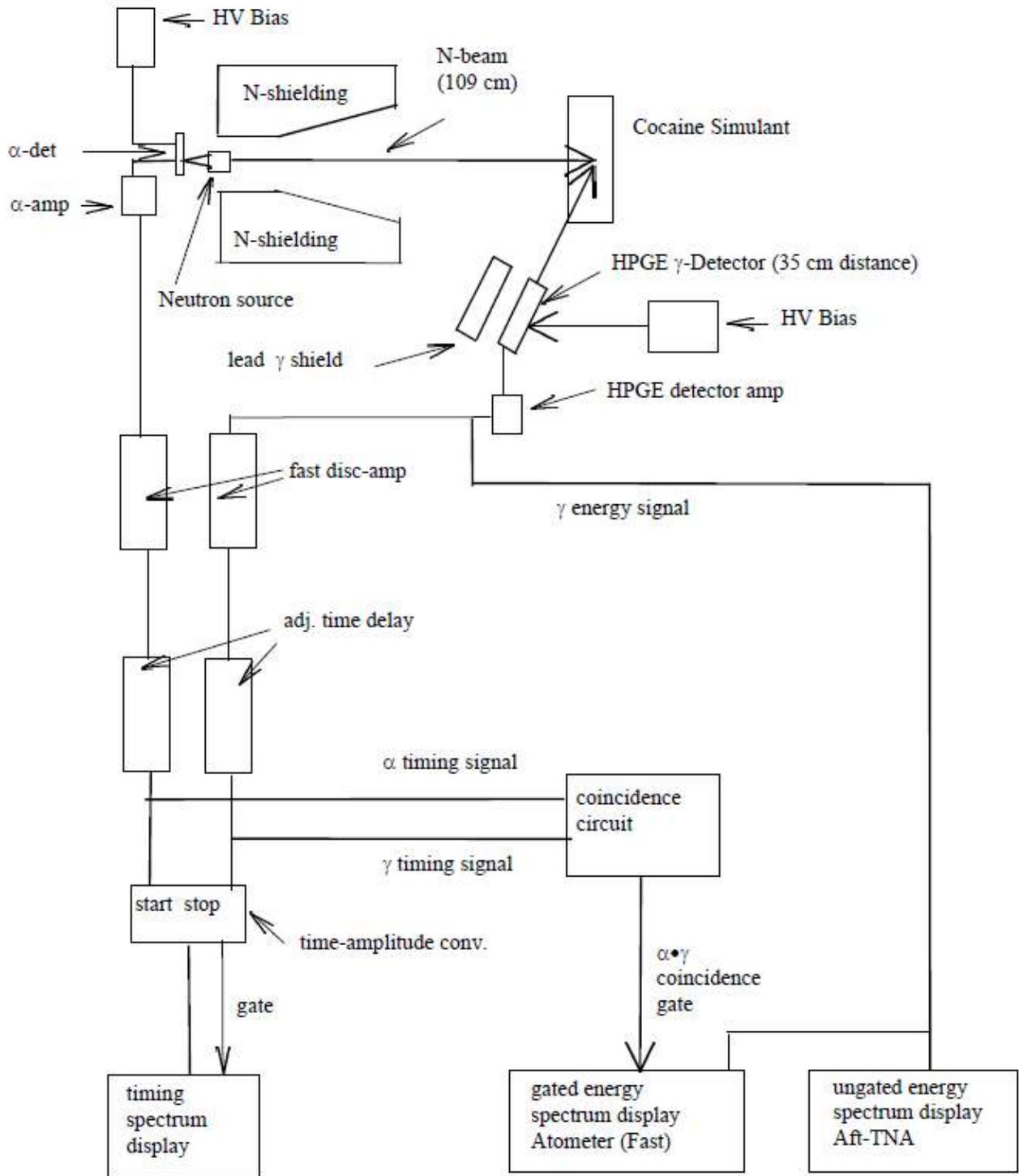
Fig. 7. Atometer 1: Diagram of experimental setup and generalized schematic diagram of detection system electronics

window (6 nanosec in this case) this circuit generates a gating pulse which is sent to a multichannel

analyzer- computer system (ORTEC DSP and Pentium PC) which is also receiving the ungated nergy

signal as described in 1). The system is configured so as to only record the energy spectrum from hose

g-rays that are being analyzed when this gating pulse is present. Therefore only the g-rays created by

the "tagged" neutrons are recorded.

3): The timing spectrum, which gives the number of g 's detected versus time

after a particle detection. This is accomplished electronically by sending the a and g timing pulses

discussed in (2) to the respective start and stop input gates of a time-to-amplitude converter (TAC).

The TAC generates an output pulse whose amplitude is proportional to the time difference between the

start and stop pulse arrival time. This signal is recorded by yet another multichannel analyzer-computer

system which produces the timing spectrum. This information is useful in that it enables us to see

where in time (and thus in distance) the g-rays are being produced whether it is in the target

zone or not (in fact, many g-rays were found to be produced by neutrons interacting with the laboratory

rear wall over 3 meters away). If we gate this spectrum with the output gate of the coincidence circuit

described in 2) we can see in time where the coincidence timing window is set, which is very helpful in

aligning the gated system.

The information obtained by all the above systems can then be recorded on computer disks to make

off-line analysis of the experimental results effective, and will help us greatly when we go on to use

the full imaging system configuration in the near future.

6. Discussion

The correct formula of cocaine is $C_{21}H_{22}N_1O_4Cl_1$. The cocaine simulant used in this experiment had

the formula $C_{16}H_{14.7}N_1O_{3.16}Cl_{0.67}$ normalized to nitrogen = 1. Using advanced FNA technique devised

in 1998 and named Atometry, HiEnergy Microdevices, Inc., obtained $C_{16.8}N_1O_{3.25}H_{14.7}Cl_{0.67}$ as the

formula for the cocaine simulant.

A source of error in atometric calculations which are dominated by carbon is the intensity

measurement of the carbon peak. The carbon peak at 4.440 MeV is typically between 65 - 85 KeV

wide -- a recoil during the (n,g) inelastic collision produces a short-lived energy state. As a result,

gamma information from the carbon peak is more subjective than the clearly defined sharp peaks of

nitrogen and oxygen.

During this experiment, gamma intensity information from carbon was obtained by averaging four
measurements (Table 3). Although this eliminates ambiguity, it does so without regard to overlap of
extraneous signals in the 65 - 85 KeV range. One solution to this problem is to seek out a high
efficiency detector . A 130% efficient (relative to NaI) HPGe detector would allow the use of the
second level energy peak at 7.65 MeV without the constraints of count loss due to efficiency at high
energies which plague the current 80% efficient HPGe.

Data from this experiment not only supports the premise of atometry but it also provides a benchmark
for extrapolation of scanning times (Table 4). Ultimately, atometry will be used in a field environment
with consideration for detection time and radiation safety.

Two factors lead to reduced detection time -- increased neutron flux and advanced equipment.
Atometry projections manifest a minimum of 40 fold improvement in detection time based on
increases/advances in the following (equipment related):

1. Efficiency of a detector (100% instead of 25%)   gain of a factor of 4
2. Number of HPGe (5 instead of 1)   "   5
3. Minimum number of g's (50% of gated g's in Test 1)   "   2
4. Total   "   40

A 40 fold reduction in the integrated number of neutrons required for atometry is feasible without
increasing the neutron flux. This projection is conservatively based on electronics, hardware, and
material based on detection of 10 Lbs of drugs. For example, the data in run 128 was collected over
73,670 seconds with 4.46×10^{11} integrated neutrons. With Atometer II (32 fold collection efficiency
built in), the total integrated number of neutrons required would be $4.46 \times 10^{11}/40 = 1.12 \times 10^{10}$. At a
neutron flux of 3×10^8 s-1 4p, that would amount to a collection time of only 37 seconds.

Table 3
Events at Energy summary (from *Maestro)*

Run	Element/Energy	Net gamma events	Error (±)	AVG (±) Std. Dev
128	Carbon 4.440	1399	106	
		966	95	857± 45.3
		647	40	
		414	105	
	Nitrogen 5.104	41	6	
		32	8	30 ± 4.5
		18	9	
	Oxygen 6.129	80	13	
		88	15	85 ± 7.9
		87	13	
138	Carbon 4.440	2648	148	
		1497	166	1468± 10.4
		1119	142	
		609	98	
	Nitrogen 5.104	62	11	
		69	14	58.3± 7.6
		44	14	
	Oxygen 6.129	133	20	
		189	17	166± 11
		175	20	

A more realistic approach to Atometer II as a field unit would yield an improvement factor of 800 by
the following parameters:

1. Efficiency of a detector (100% instead of 25%)	gain of a factor of 4
2. Number of HPGe (10 instead of 1)	" 10
3. Increase cocaine mass (100 Lbs of drug)	" 10
4. Minimum number of g's (1/2 # of gated g's)	" 2
5. Total	" 800

Table 4

Measured parameters of experimental runs with Atometer 1 (low performance system)and projected parameters for Atometer 2 (bottom line).

Run #	Target Description	Time (s)	Total Alpha's	Mass (Kg)	Total Neutrons	Flux ($4\pi s^{-1}$)
126	Background	63,040	2.46×10^9	0	3.63×10^{11}	5.76×10^6
127	Urea	81,830	4.16×10^9	5	6.10×10^{11}	7.46×10^6
128	(Run D)Cocaine Simulant	73,670	3.04×10^9	4.54	4.46×10^{11}	6.05×10^6
138	(Run D)Cocaine Simulant	105,800	4.34×10^9	4.54	6.37×10^{11}	6.02×10^6
132	(Run A) 30 LBS. Rice	15,240	9.35×10^8	11.8	1.37×10^{11}	9.00×10^6
134	(Ruc B)Cocaine sim. + Rice	69,560	3.50×10^9	16.3	6.45×10^{11}	9.29×10^6
Atom-eter 2	Cocaine simulant	50	1×10^8	4.54	1.6×10^{10}	3.0×10^8

Therefore, with Atometer 2 (800 fold collection efficiency built in), the total integrated number of neutrons required would be $4.46 \times 10^{11}/800 = 5.57 \times 10^8$. At a neutron flux of 3×10^8 s-1 4p, that would amount to a collection time of 1.9 seconds(i.e. 100 Lbs of drug [1.1 meter away] could be chemically identified to within 98% accuracy in 1.9 seconds).

With the current electronics circuit diagram of Atometer 2, it is possible to use a neutron flux of 3×10^9 s-14p. This is a technological advantage to lowering radiation levels because it would give an operator a tool for a rapid "scan" only if deemed necessary.

The other remaining concern for the use of atometry in the field is radiation safety. Our projections (U. of Kansas) yield that with an average neutron flux of 3×10^8 s-14p, with 80x80x80 cm3 of borated heavy polyethylene as neutron shielding, an operator would receive a total annual dose of 0.04 rem (2,000 hours). This factor could be lowered by an order of magnitude with every additional 25 cm3 of shielding.

7. Conclusions

1. Accurate empirical chemical formula of cocaine core (CNO) was retrieved by single arm Fast Neutron Atometry.
2. The retrieval time with a neutron source delivering 3x10(8) [3x10(9)] n/s/4pi is 50 [5] sec.
3. Atometry meets and exceeds all the requirements of the US Customs Service in terms of low false alarm rates based on 95% accuracy, fast detection times (1.9 seconds with a neutron flux of 3×10^8 s-14p) and low radiation hazard (0.004 rem/year with 105x105x105 cm3 cube shielding).
4. Accurate empirical chemical formula of cocaine in rice was retrieved by a single arm Fast Neutron Atometry for rice/cocaine mass ratio of 2:1.

5. The empirical chemical formula of cocaine was not obtained for rice/cocaine mass ratio 3:1. It is inconclusive of this result is intrinsic or accidental.

6. The fast C/O ratio is significantly higher for both rice/cocaine ratii, 2:1 and 3:1 and it can serve as a prompt drug detector.

7. The detection sensitivity threshold will be increased by an order of magnitude by replacing the bulk imaging with, at least, 128 alpha imaging element.

Fig. 2

Dalitz Plot --Differential Atometry -- Cocaine embedded in Rice

Carbon

Oxygen

C -- corrected for absorption

A

C

C, D

Nitrogen

• Simulant   • Rice
■ Rice+Cocaine   • R + Cocaine ABS
□ IR+Cocl-Rice

Fig 3.

Time of Flight (0.29 ns/ch)

C B A

◆ 30 LBS Rice + 10LBS Cocaine ■ 10 LBS Cocaine

Geometry:
10 LBS. of rice (Gray) is infront of rice with width of 10 cm or 7 channels (A) centered at 566;
10 LBS. of cocaine (Orange) with width of 10 cm or 7 channels (B) centered at 559;
10 LBS. of rice (Gray) behind cocaine with width of 10 cm or 7 channels (C) centered at 552;
The 22 channel gate was centered on channel 559 corresponds to a gate of 6.38 ns or 32.5 cm.

Wall

Counts

Time

Channel #