J. Pharm. Pharmacol. 1992, 44: 1–6
Received May 2, 1991

Illicitly Imported Heroin* Products (1984 to 1989): Some Physical and Chemical Features Indicative of Their Origin

PHILIP J. O'NEIL AND JANE E. PITTS

Laboratory of the Government Chemist, Teddington, Middlesex, UK

Abstract—Samples taken from seizures of imported illicit heroin preparations of known geographical origin have been examined. The typology developed in two previous surveys of illicit heroin products is applicable to many of the samples studied in this work, although significant changes have occurred in the chemical profile of illicit heroin products from certain geographical regions. It remains possible, however, to give an opinion as to the origin of many samples of illicit heroin of unknown provenance. The observation in the previous surveys that unrelated samples of illicit heroin possess unique chemical profiles has been confirmed by the present results.

The relationship between the geographical origin of illicit heroin samples and their resulting physical appearance and chemical composition is well established (O'Neil et al 1984; O'Neil & Gough 1985). This paper presents physical and chemical data on 923 illicit heroin samples of known country of origin. The samples in this study represent by quantity most of the illicit heroin seized by Officers of Her Majesty's Customs and Excise in the period January 1984 to December 1989. Two types of illicit heroin are excluded—samples smuggled within body orifices of the courier ("body packers", "stuffers and swallowers") and samples that had been impregnated into an article. Body packed illicit heroin is excluded because laboratory autoclaving to make it biologically safe could affect the heroin:6-acetylmorphine ratio. Impregnated illicit heroin is excluded because, although a chemical profile can be obtained, it is impossible to determine concentrations in the original powder. Body-packed and impregnated samples are comparable with conventionally smuggled illicit heroin from the same country. Their exclusion, although reducing the number of profiles, has little effect on trends and conclusions drawn.

Even after exclusion of impregnation and stuffer and swallower samples, the heroin included in the study represents over 75% by quantity of the total heroin seized by UK law enforcement agencies in the period 1984 to 1989. In the period of this study smuggling routes to the UK were considerably diverse. Much of the illicit heroin entering the United Kingdom in this period had passed through one or more transit countries.

Since this paper covers a five year time scale we have been able to examine the change from year to year in the average heroin content of samples of a common origin. We have restricted this to the three main exporting countries, Pakistan, India and Nigeria, because the number of samples per year from other regions is insufficient to draw firm conclusions about changes in opiate content. However, the limited samples from other regions have shown no significant change in opiate content over the period of this study.

*In this paper "heroin" means pure diacetylmorphine. "Illicit heroin" means impure diacetylmorphine that contains related narcotics and other materials and is of clandestine origin.

Correspondence: J. E. Pitts, Laboratory of the Government Chemist, Queens Road, Teddington, Middlesex TW11 0LY, UK.

Experimental Procedure

The procedure adopted for taking representative samples of illicit heroin was that described by O'Neil et al (1984) and O'Neil & Gough (1985). Virtually all samples were taken from seizures or batches between 250 g and 2 kg. Many large importations consisted of multiple samples each weighing 1 kg. The origin assigned to samples in all three papers is the country from which the heroin was exported to the UK. We stress that this may not be the producing country, indeed in the case of one exporting country, Nigeria, it seems doubtful that any indigenous heroin is produced. Samples in this survey have been analysed using the HPLC method described by Huizer (1978) with some variation in the mobile phase. GCMS was used to identify some unknown components which were invariably non-opiate adulterants.

Solutions for analysis

The solutions contained 15 mg in 25 mL of solvent of the same composition as the mobile phase.

Apparatus

The pump was a Waters M45 operating at 1500 psi. The detector was a Spectra Physics 8450 UV/Vis detector. The operating conditions were: column, Lichrosorb Si-60 (Technicol) in 250 × 4 mm stainless steel; 20 μL injection with a Waters Intelligent Sample Processor (WISP); detection, ultraviolet at 227 nm. The whole system was controlled by, and data collection carried out on, a Waters 840 datastation, which was run on a DEC Professional computer. Up to 90 samples could be analysed in one run.

The mobile phase (hexane, dichloromethane, methanol (containing approx. 4% diethylamine), 75:20:5) achieved baseline separation of the five opiates (narcotine, papaverine, acetylcodeine, heroin and 6-acetylmorphine) and the three commonly encountered adulterants (methaqualone, caffeine and phenobarbitone). Due to the large number of samples containing other adulterants such as *N*-phenylnaph-

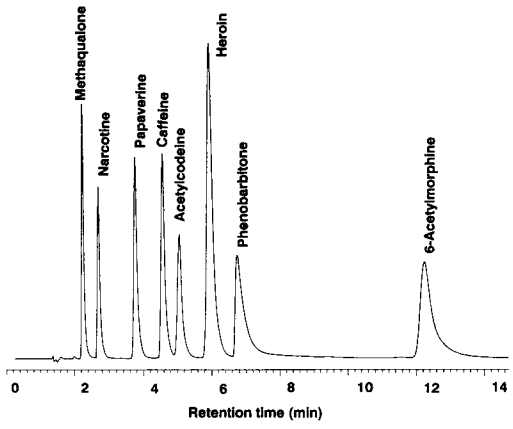

FIG. 1. HPLC separation of putative components of illicit heroin samples.

thylamine, procaine, and niacinamide, it was necessary to adjust further the amount of diethylamine in the mobile phase to obtain baseline separation. This affected retention times but not the elution order of the eight common components (Fig. 1).

Results and Discussion

The number of samples seized from Pakistan, India and Nigeria per year of this survey are noted in Table 1. Full results are available from the authors but a resumé of our finding is now given.

Table 1. Number of samples seized 1984 to 1989.

Year	Pakistan	India	Nigeria
1984	74	33	—
1985	32	67	—
1986	16	71	18
1987	63	38	12
1988	88	32	11
1989	73	45	84

Compared with pre-1984 the heroin entering the UK in 1984 to 1989 was a much more complex material. In the period of this study large consignments of heroin were most often found to be composed of a number of different batches, each with its own unique chemical profile. The chemistry of each batch was often further complicated by the presence of one or more adulterants. Multi-batch importations are both a disadvantage and an aid to the forensic chemist seeking to link individual seizures. It is a disadvantage because once such consignments are divided a chemical connection between the batches may not be possible. It is an aid because the evidential value of a positive comparison of seizures is in proportion to the number of different batches identified. In addition there is the possibility that separate seizures, each composed of more than one batch of heroin, can be multiply linked.

Physical appearance

For many samples the physical appearance bears little or no relationship to the heroin content. All the base samples were brown powders some of which had been loosely compressed. Although there was a wide variation in shades of brown, many samples did have a common colour; this colour closely matches that indicated in the Colour Handbook (1978) as 5C4 and 6C4.

With a few exceptions, all samples containing heroin hydrochloride were white or off-white powders. Some had been heavily compressed into slabs; chemical analysis showed these to be "Chinese No. 4".

Chemical composition

The average opiate contents of the base and hydrochloride samples, by country, are listed in Table 2.

Illicit heroin from SW Asia (Pakistan and India)

Pakistan (346 samples) and India (286 samples) are the two major exporting countries in this study (Table 1), most of the samples being in the base form. During the period 1984 to

Table 2. Average opiate content (%) by country and sample type. Only the main countries and types are presented.

Country/region	Type of sample	Heroin	Acetylcodeine	6-Acetylmorphine	Noscapine	Papaverine
SE Asia	"Chinese No. 4"	81·26	7·78	2·62	NP[1]	NP
Turkey	All samples[2]	33·60	3·28	3·64	20·26	1·58
Nigeria	"SW Asian" (all samples)	36·80	2·39	2·49	19·09	1·28
	Unadulterated[3]	41·07	3·58	3·29	19·16	1·46
	Adulterated	36·21	2·23	2·38	19·07	1·25
	"SE Asian"	77·39	7·32	3·87	NP	NP
Pakistan[4]	Base heroin (all samples)	43·13	2·89	2·44	16·23	1·32
	Unadulterated	49·43	3·85	3·52	14·80	1·49
	Adulterated	30·39	2·26	1·72	17·17	1·21
	HCl heroin	66·75	2·04	7·47	1·35	0·03
India[4]	Base heroin (all samples)	41·66	3·08	2·53	15·48	1·29
	Undulterated	50·66	3·66	2·66	17·03	1·51
	Adulterated	33·08	2·51	2·40	14·00	1·08
	HCl heroin	18·73	0·70	2·35	NP	NP

[1] NP = Not present (although a few samples contained trace quantities). [2] One untypical sample excluded. [3] In this table adulteration means the addition of any non-opiate compound. [4] SW Asian base heroin "Fakes" (q.v.) excluded from averaging.

1989 illicit heroin from SW Asia has undergone major changes in comparison with samples imported into the UK before then.

A classification differentiating the SW Asian base samples by country cannot be developed for the period. Base samples from both countries have to be considered as a single product, although some differences can be seen. The chemical similarity of base samples from both countries is confirmed by law enforcement intelligence that much illicit heroin crosses the Indian/Pakistan border and that a sample, ostensibly from one SW Asian country, may have a true origin elsewhere in SW Asia.

The major change during this period was the frequent presence of adulterants in base samples. The effects of adulteration on the heroin content for both Pakistan and India base samples are shown in Figs 2 and 3, respectively, while the frequency of adulteration is shown in Fig. 4. Although there are some differences in the pattern of adulteration of base samples from Pakistan and India, these are still insufficient to enable adulterated samples from the two countries to be classified as distinct products.

Adulteration of SW Asian base samples began tentatively in early 1984 but the compounds subsequently to dominate as adulterants were rare in 1984 samples. Frequent adulteration for both Pakistan and Indian samples began in the second half of 1985; by the end of 1985, for both countries, the pattern of adulteration was set: methaqualone, phenobarbitone and caffeine would predominate while other compounds, overwhelmingly pharmaceutical, would be spasmodically detected. Samples containing caffeine, or methaqualone and caffeine, or all three cutting agents, were more common from Pakistan (Table 3); samples containing methaqualone, phenobarbitone or both were more common from India (see Table 4).

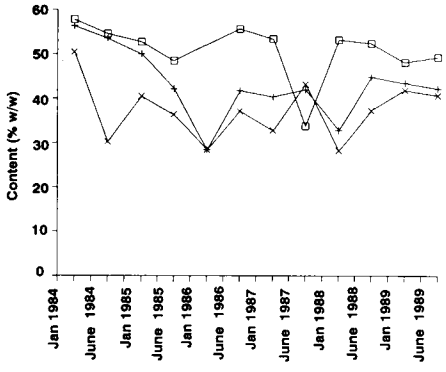

FIG. 2. Heroin content of illicit heroin samples from Pakistan, 1984–1989. +, all samples; □, non-adulterated; ×, adulterated.

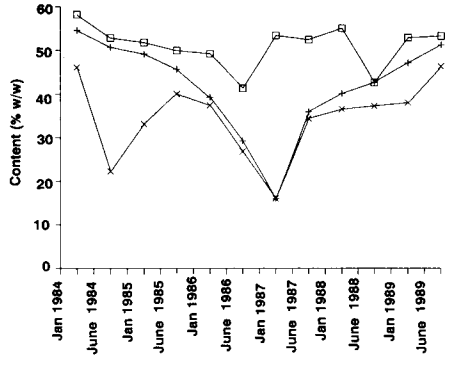

FIG. 3. Heroin content of illicit heroin samples from India, 1984–1989. +, all samples; □, non-adulterated; ×, adulterated.

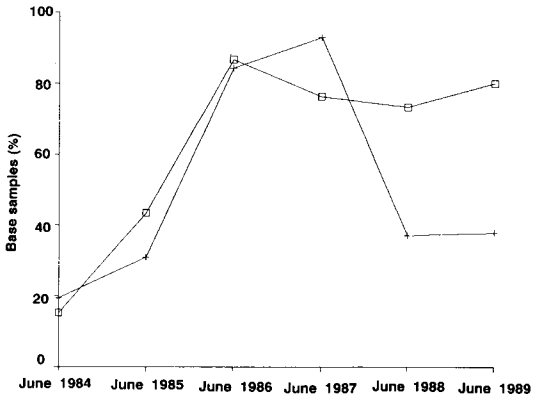

FIG. 4. Frequency of adulteration of illicit heroin samples from India (+) and Pakistan (□).

From 1984 the heroin content of adulterated Pakistan samples declined. Between 1986 and 1988 it remained reasonably constant, being typically 30–40%. The decline of the heroin content of Indian adulterated samples was greater than those from Pakistan reaching a nadir during the first half of 1987; between 1986 and 1988 Indian adulterated samples had a typical heroin content of 15–35%.

The heroin content of adulterated samples from SW Asia recovered towards the end of the 1980s although it did not regain the high level of 1983 (O'Neil & Gough 1985). The

Table 3. Pakistan base samples and their most common adulterants.

		%
Methaqualone + caffeine + phenobarbitone	59	18·6
Methaqualone + caffeine	24	7·6
Caffeine + phenobarbitone	24	7·6
Methaqualone + phenobarbitone	10	3·1
Methaqualone only	21	6·6
Caffeine only	35	11·0
Phenobarbitone only	08	2·5
Total adulterated samples	181	57·1
Total unadulterated samples	136	42·9
Total samples	317	100

Table 4. Indian base samples and their most common adulterants.

		%
Methaqualone + caffeine + phenobarbitone	33	12
Methaqualone + caffeine	05	1·8
Caffeine + phenobarbitone	20	7·3
Methaqualone + phenobarbitone	16	5·8
Methaqualone only	34	12·4
Caffeine only	09	3·3
Phenobarbitone only	18	6·5
Total adulterated samples	135	49·1
Total unadulterated samples	140	50·9
Total samples	275	100

recovery is noteworthy because the heroin content of non-adulterated samples from SW Asia was meanwhile declining (due to higher levels of noscapine, see below).

By 1989 adulteration had become nominal and, in contrast to 1985 to 1988, it was rare for the content of any non-opiate adulterant to exceed 10%.

Other adulterants. Chronologically, paracetamol was the first adulterant, being detected during 1984 in Pakistan and Indian samples. Phenacetin, common in early Indian samples, occurred in only two Pakistan samples. Niacinamide, common in recent Pakistan samples, occurred less frequently in Indian ones.

Noscapine and papaverine. The noscapine content of SW Asian base samples increased from 1984 to 1989 but the papaverine content of the same samples did not rise. Some 1989 unadulterated samples were found to contain over 30% noscapine, and in excess of 20% was normal. Annual average noscapine and papaverine contents are shown in Figs 5 and 6, respectively.

There are two possible explanations for the increased noscapine content. First, a different method (ammonia rather than lime) may have been used to extract morphine from opium. This method, without a stage to remove noscapine and papaverine before the precipitation of the morphine, results in higher impurity levels. Second, pre-precipitated noscapine may have been added to synthesized

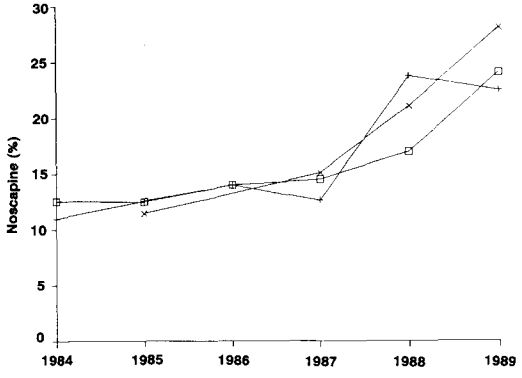

FIG. 5. Increase in noscapine content of all base heroin samples from India (+), Pakistan (□) and Nigeria (×), 1984–1989.

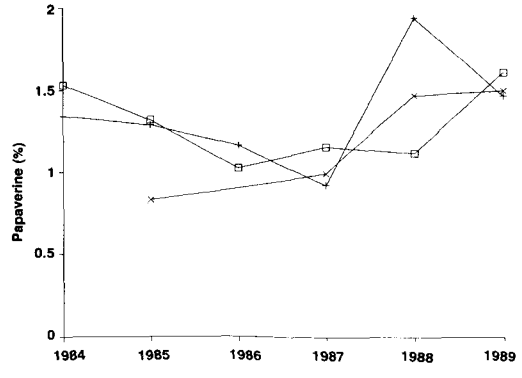

FIG. 6. Increase in papaverine content of all base heroin samples from India (+), Pakistan (□) and Nigeria (×), 1984–1989.

illicit heroin. Recent samples in the authors' laboratory and in Holland (Huizer, personal communication) were found to contain distinct particles of noscapine, lighter in colour than the bulk of the powder. Either explanation would result in a greater mass of powder being produced.

By contrast three base samples all from India contained no noscapine or papaverine, and other recent Indian samples contained no 6-acetylmorphine.

Fakes. 13 base samples from Pakistan and 14 from India were found to contain little or no heroin. Of the 13 from Pakistan, 4 contained no heroin, 3 less than 1% and 6 less than 2%; none of the Indian samples were devoid of heroin, 3 contained less than 2%, 7 had 2–5%, and 4 had 5–10%. The samples contained no morphine and little 6-acetylmorphine and therefore are not ones in which acetylation had not been fully realised, nor ones in which the heroin had subsequently decomposed. We have excluded these from the statistical analysis of purity levels. They are fakes in a trafficking context because they were major smuggling quantities (where the trafficker assumes a reasonable heroin content) rather than small street-level samples (where a buyer may expect low heroin levels). Physically the fakes closely resembled SW Asian base heroin, the material consisting of combinations of the common adulterants and opiate alkaloids, probably the remnants of heroin synthesis.

Hydrochloride samples from SW Asia. In the period 1984 to 1989, unlike the base samples, hydrochloride samples from Pakistan and India could be differentiated.

Sixteen of the 346 samples from Pakistan, 9 of the 286 samples from India and 1 from Nepal contained heroin hydrochloride. Most of the Pakistan samples had heroin contents comparable with "Chinese No. 4" but were easily differentiated by their lower acetylcodeine contents, higher levels of 6-acetylmorphine (poor synthesis), and residual noscapine and papaverine contents (poor extraction of morphine from opium). No adulterants were detected in the Pakistan samples. All the Indian samples were characterized by low opiate levels; none had a heroin content above 30%, or an acetylcodeine content over 1·3%, and all were devoid of noscapine and papaverine. The bulk of material in every Indian sample was either lactose or mannitol.

Illicit heroin from SE Asia

Only 50 illicit heroin samples originated from SE Asia in the period 1984 to 1989. Virtually all of these were of "Chinese No. 4". For the first time, some samples (all from Singapore) were found to contain high levels of 6-acetylmorphine and morphine. Reanalysis of these samples five years later showed little changes in the heroin/6-acetylmorphine/morphine ratios. This suggests that poor manufacture rather than post-synthesis decomposition is the reason for the presence of 6-acetylmorphine and morphine.

The only adulterant detected in SE Asian illicit heroin was caffeine, present in 5 of the samples. Three samples were chemically and physically very similar to SW Asian illicit heroin. Some samples, although essentially "Chinese No. 4", contained detectable levels of noscapine. In all but one of these, however, the papaverine had been entirely eliminated.

SE Asian illicit heroin continues to have a high acetylco-

deine content; 8 have acetylcodeine contents over 10%. With very few exceptions these samples had low levels of 6-monoactylmorphine and no morphine, indicating good manufacturing technique.

Illicit heroin from Nigeria

In terms of number of cases and quantity of drug Nigeria is the third major supplier of illicit heroin to the UK. There are no reports of poppy growth or opium production in Nigeria; chemically and physically, samples exported from Nigeria can be assigned an origin in either of the two main heroin producing regions of the world: SW or SE Asia. Law-enforcement intelligence reports confirm these findings.

Of the 125 Nigerian samples analysed, 84 contained heroin base and 43 heroin hydrochloride. The opiate chemistry of the 84 base samples was comparable with that of SW Asian base heroin and, in addition, these samples had a high frequency of adulteration. Only 10 of the 84 samples had no adulterants. The adulterants detected, the pattern of adulteration, and the concentrations of the adulterants were all very similar to SW Asian base heroin.

From 1986 to 1989 the heroin content of Nigerian base samples had an increase comparable with that of SW Asian base samples. The increase in the noscapine content in SW Asian base samples was also duplicated by Nigerian base samples, but the papaverine content of Nigerian samples also increased, albeit less markedly than noscapine (See Figs 5, 6, 7). The change in relative proportions of the opiates, unlike SW Asian base samples, is probably because of the ammonia extraction method and not the addition of extra noscapine. Given the physical and chemical similarities, SW Asia is the likely origin of Nigerian base samples.

During the period of this study, Nigerian base samples were the most frequently adulterated heroin products being imported into the UK (Table 5).

Methaqualone, phenobarbitone and caffeine were the most common adulterants but some of the less common adulterants found in SW Asian heroin were also detected in Nigerian base samples.

Assignment of the likely country of origin within the Indian subcontinent for the Nigerian base heroin samples is speculative and any conclusions can only be made globally, i.e. there are exceptions to any classification developed and the country of origin of any Nigerian base sample cannot be determined with any confidence. Examination of Tables 3, 4 and 5 reveals more correlations between Nigeria and Pakistan than between Nigeria and India.

Table 5. Nigerian base samples and their most common adulterants.

		%
Methaqualone + caffeine + phenobarbitone	21	25·0
Methaqualone + caffeine	22	26·2
Caffeine + phenobarbitone	9	10·7
Methaqualone + phenobarbitone	2	2·4
Methaqualone only	10	11·9
Caffeine only	10	11·9
Phenobarbitone only	0	0
Total adulterated samples	74	88·1
Total unadulterated samples	10	11·9
Total samples	84	100

Hydrochloride samples from Nigeria. The high average levels of acetylcodeine in the 43 hydrochloride samples suggests SE Asia as their most likely origin. Of the 43 samples only 3 had acetylcodeine levels below 2% and these 3 are uncharacteristic samples in that their heroin content was only 25 to 40% and the levels of the other opiates were low. Excluding these 3 samples produces an average acetylcodeine content of 7·76% which compares well with the average acetylcodeine content of 7·78% for "Chinese No. 4" heroin (Table 2). Three of the hydrochloride samples contained unusually high levels of acetylcodeine (approx. 25% by weight). Confirmation of the acetylcodeine content was obtained by GC (O'Neil et al 1984) and the samples were screened by TLC (United Nations 1986) to determine if a co-eluted component was causing the high values. Both techniques confirmed the HPLC results. As with SE Asian hydrochloride samples, a small number of the Nigerian hydrochloride samples were found to contain quantifiable amounts of noscapine although all were devoid of papaverine.

Illicit heroin from Africa (other than Nigeria)

Of the 19 African samples, 14 came to the UK from West Africa and 5 from Central/East Africa. All the exporting countries have trading links with SW Asia. Chemically and physically, illicit heroin from Africa was similar to SW Asian heroin.

Seventeen out of 19 of the samples had the typical chemistry of base heroin samples from SW Asia; most had been adulterated with the combinations of methaqualone, phenobarbitone and caffeine. Two samples contained heroin hydrochloride adulterated with mannitol; these strongly resemble samples known to originate in India.

Illicit heroin from Turkey (including Turkish Cyprus)

Few illicit heroin samples from Turkey were detected in the UK from 1984 to 1988 and of the 71 samples in this study 51 were seized in 1989. The samples from Turkey and Cyprus fall into two groups. Samples in the first group contain procaine, an adulterant not encountered in any sample from any other country or region (O'Neil et al 1984; O'Neil & Gough 1985). The second group of samples was chemically similar to SW Asian but the frequency of adulteration in Turkish base samples was much lower than in SW Asian base samples. The adulterants detected, however, were those common in SW Asian samples: methaqualone, phenobarbitone, caffeine and, in 1989, noscapine. One large importation consisted of a multi-batch consignment of heroin from both groups. Our analysis of the samples from the second group suggests, therefore, that Turkey is a transmitting country for heroin from SW Asia. Only four samples, all dating from 1984 to 1986, contained heroin hydrochloride. Twenty-two samples, however, were found to contain morphine, previously undetected in Turkish samples.

Most of the base samples had a heroin content between 12 and 55% which is the typical range of purity for Turkish samples (O'Neil et al 1984; O'Neil & Gough 1985). As expected, the average heroin content of the samples containing morphine was lower (25·22%) than that of samples without morphine (37·53%).

Because most Turkish samples are from 1989 it is not meaningful to monitor annual average opiate levels; the

average heroin content for 1989 samples (30·11%) was lower than the average for samples from 1984 to 1988 (43·50%) but this is because all the samples containing procaine or morphine were from 1989. The noscapine content of Turkish samples, like SW Asian base samples, increased towards the end of the 1980s.

Illicit heroin from the Near East and Iran

Two characteristics of Near Eastern illicit heroin previously noted were the frequent presence of high levels of caffeine (other adulterants were unknown) and that all samples were hydrochlorides. In this study all five Lebanese samples contained caffeine and three were hydrochlorides. The two most recent Lebanese samples, however, were base ones, and were the only illicit heroin samples in this study that contained more papaverine than noscapine.

One of the three Iranian samples contained caffeine and phenobarbitone, and was indistinguishable from adulterated SW Asian base heroin. The two other samples, part of the same consignment, were high purity hydrochloride ones.

Illicit heroin from Europe

These include samples seized within the UK for which the country of export could not be found, and samples whose smuggling route could only be traced as far as a European country. The samples from Europe are typical unadulterated SW Asian base. Of the samples seized within the UK two contained methaqualone and were probably from SW Asia, while the high caffeine content of two others suggested the Middle East (Lebanon, Syria) as their origin. Six samples, all containing chloroquine and probably of Near Eastern origin, were intercepted as a single detection at export from the UK to the USA. The chloroquine content was obtained by PMR spectroscopy.

References

O'Neil, P.J., Baker, P.B., Gough, T.A. (1984) Illicitly imported heroin products: some physical and chemical features indicative of their origin. J. Forens. Sc. 29: 889–902

O'Neil, P.J., Gough, T.A. (1985) Illicitly imported heroin products: some physical and chemical features indicative of their origin: Part II. Ibid. 30: 681–691

Huizer, H. (1978) Analytical studies on illicit heroin: comparison of samples. Ibid. 28: 40–48

Kornerup, A., Wanscher, J.H. (1978) The Colour Handbook. Eyre Methuen, London

United Nations (1986) Recommended Methods for Testing Heroin